Pilot mechanical pulping assessment of dry blue-stained and grey-stage wood chips from beetle-killed lodgepole pine

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Mountain Pine Beetle Working Paper 2008-25

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Abstract

Pilot-plant thermomechanical (TMP) pulping has been performed on green and dry, blue-stained grey-stage lodgepole pine (LPP) chips with different moisture contents. The energy requirements for TMP pulping of the dry, early-grey (kill date approximately 4 years) chips and the dry, late-grey (kill date approximately 6 to 8 years) chips were similar to those for TMP pulping of the green chips, when all the results were taken into consideration. The early-grey and late-grey TMP pulps had lower sheet density and ISO brightness, and higher handsheet surface roughness than the green TMP pulps. No significant difference in long-fibre and fines contents, fibre-length or tensile strength was observed between the green and the early-grey TMP pulps at the same freeness, while indications of a slightly lower tensile index were found for the late-grey TMP. The late-grey TMP also showed a higher light scattering coefficient at a given Canadian Standard Freeness (CSF).

The higher handsheet surface roughness of the late-grey and early-grey TMP pulps was not due to the low moisture content of the late-grey and early-grey chips, but likely due to other changes. The maximum level of the dry, late-grey chips that could be added to the green chips without significantly increasing the surface roughness or lowering the sheet density was 25%.

Low-intensity refining in the post-primary stages of the early-grey and the late-grey TMP improved the fibre length, but did not affect the tensile index at a given CSF significantly. Chip presteaming and water impregnation had a marginal effect on the pulp properties of the late-grey TMP. However, sodium sulfite pretreatment at pH=6 coupled with low-intensity refining of the late-grey chips had a positive effect on the tensile index at a given CSF. Process modifications involving careful sulphite pretreatment could be investigated further if loss of tensile strength due to beetle-killed wood becomes a greater problem in the future. Reducing the refining intensity may be a solution if maintaining the fibre length becomes a critical issue. Both these modifications led to somewhat increased refining energy consumption to reach a given CSF.

Increase of moisture content/water impregnation of the early- and late-grey LPP chips could be achieved by increasing chip presteaming temperature/pressure and/or time, by increasing the water impregnation time, or by controlling the conductivity and pH of the impregnating water at ~200 μS/cm and ~6.0, respectively. The most economical pretreatment approach to increasing the moisture content of the dry, grey-stage LPP chips prior to TMP pulping appeared to be presteaming at moderate temperature/pressure, followed by 8 to 10 minutes of chip washing with proper control of the conductivity and pH of the chip washing water.

Soaking of the green and the late-grey blocks in a 10% $AgNO_3$ solution, followed by ESEM-EDS analysis of Ag intensity, could provide qualitative information on the movement of water within 10 minutes of water soaking/impregnation. ESEM-EDS results on the green and late-grey blocks are consistent with the water-uptake results obtained using Weyerhaeuser test apparatus. Dry late-grey LPP chips behaved very

differently during TMP pulping or water/chemical impregnation than green LPP chips air-dried to the same starting moisture content.

Keywords: grey-stage, fibre properties, mountain pine beetle, blue stain, mechanical pulps, moisture content, pulp strength, sheet properties, surface properties

Résumé

De la pâte à papier a été fabriquée à l'aide d'un procédé thermomécanique dans une usine-pilote (TMUP) à partir de copeaux verts, secs et bleuis de pin de Murray au stade gris ayant un taux d'humidité variable. Les besoins énergétiques de la fabrication de pâte à papier à l'aide du procédé TMUP à partir de copeaux secs au début du stade gris (détruits il y a environ quatre ans) et de copeaux secs à la fin du stade gris (détruits il y a environ six à huit ans) étaient similaires à ceux de la fabrication de pâte à papier à l'aide du procédé TMUP à partir de copeaux verts, tous résultats confondus. La densité des feuilles fabriquées à partir de copeaux au début et à la fin du stade gris était inférieure, de même que la brillance ISO. En outre, la surface de la feuille d'essai était plus rugueuse que celle fabriquée à partir de copeaux verts. Aucune différence significative n'a été observée dans le contenu des fibres longues et des particules inclassables, la longueur des fibres ou la résistance à la traction des pâtes à papier fabriquées à partir de copeaux verts ou au début du stade gris à indice d'égouttage identique. Toutefois, on a constaté que l'indice de traction de la pâte fabriquée à partir de copeaux à la fin du stade gris était légèrement inférieur. De plus, le coefficient de diffusion de la lumière de la pâte fabriquée à partir de copeaux à la fin du stade gris était supérieur pour un indice d'égouttage donné.

La rugosité de la surface de la feuille d'essai fabriquée à partir de copeaux à la fin et au début du stade gris n'était pas attribuable au faible taux d'humidité de ces copeaux, mais plutôt à d'autres changements. Il a été possible d'augmenter le taux d'humidité des copeaux secs et à la fin du stade gris d'au maximum 25 % sans accroître de façon considérable la rugosité de la surface ou réduire la densité de la feuille.

Le raffinage à faible intensité aux étapes postprimaires du procédé TMUP au début et à la fin du stade gris a permis d'améliorer la longueur des fibres, sans toutefois modifier considérablement l'indice de traction à un indice d'égouttage donné. Le préétuvage des copeaux et l'imprégnation dans l'eau ont eu un effet insignifiant sur les propriétés de la pâte à papier fabriquée à partir de copeaux à la fin du stade gris. Toutefois, le prétraitement au sulfite de sodium avec un pH de 6 conjointement avec un raffinage à faible intensité des copeaux à la fin du stade de gris ont eu un effet positif sur l'indice de traction à un indice d'égouttage donné. Si la perte de résistance à la traction, en raison de l'utilisation de bois détruit par les scolytes, devient un problème à l'avenir, il faudrait étudier la possibilité de modifier le processus de fabrication, c'est-à-dire utiliser un prétraitement au sulfite de sodium. Si le maintien de la longueur des fibres devient une préoccupation, la réduction de l'intensité de raffinage pourrait être une solution. Ces deux

modifications se sont toutefois traduites par l'augmentation de la consommation énergétique durant le raffinage, afin d'obtenir un indice d'égouttage donné.

Il serait possible d'accroître le taux d'humidité/l'imprégnation à l'eau des copeaux au début et à la fin du stade gris du pin de Murray en élevant la température, la pression ou la durée de préétuvage des copeaux, en augmentant la durée d'imprégnation à l'eau ou en contrôlant la conductivité et le pH de l'eau utilisé pour l'imprégnation à ~200 µS/cm et ~6,0, respectivement. L'approche de prétraitement la plus économique destinée à augmenter le taux d'humidité des copeaux secs au stade gris du pin de Murray avant l'utilisation du procédé TMUP pour fabriquer de la pâte à papier est le préétuvage à température/pression modérée, suivi d'un lavage de 8 à 10 minutes des copeaux en contrôlant la conductivité et le pH de l'eau utilisée pour le lavage.

Le trempage des copeaux verts et à la fin du stade gris dans une solution de 10 % de nitrate d'argent, suivi de l'analyse au moyen de la microscopie électronique à balayage environnemental - de la spectrométrie de rayons X à dispersion d'énergie (ESEM-EDS) de l'intensité d'argent, pourraient fournir de l'information qualitative sur le mouvement de l'eau durant les 10 minutes que dure le trempage ou l'imprégnation à l'eau. Les résultats de l'ESEM-EDS pour les copeaux verts et à la fin du stade gris sont cohérents avec les résultats d'absorption de l'eau obtenus au moyen de l'appareil d'essai Weyerhaeuser. Les copeaux du pin de Murray, secs et à la fin du stade gris, n'ont pas réagi de la même façon durant le procédé TMUP ou l'imprégnation à l'eau chimique que les copeaux verts du pin de Murray, séchés à l'air en vue d'obtenir le même taux d'humidité avant le traitement.

Mots-clés : stade gris, propriétés des fibres, dendroctone du pin ponderosa, bleui, pâte mécanique, taux d'humidité, résistance de la pâte, propriétés de la feuille, propriétés de la surface.

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1 Introduction

1.1 Project rationale

Blue-stained wood chips comprise the majority of the current lodgepole pine (LPP) chip supply to British Columbia's (BC's) pulp mills. In addition to the intractable blue coloration, blue-stained wood has also been shown to exhibit moisture content at or below fibre saturation point (FSP) (24%-30% on dry wood basis, approximately 19%-23% on wet wood basis). For grey-stage mountain pine beetle (MPB)-infested LPP (time since death 4 years or longer), the material which will comprise the longer-term wood chip supply, stem moisture contents in the range of 9%-30% (wet basis) have been measured (Dalpke et al. 2007; Trent et al. 2006). This makes it of questionable utility for solid lumber applications as the loss of moisture from the cell wall is accompanied by shrinkage and defects such as internal checking. Utilization of this wood thus depends heavily on the pulp and paper industry. The wood-water interaction of beetle-killed lodgepole pine has been investigated in Radiotis et al. 2008).

Mechanical pulp producers have reported anecdotally, along with pulp brightness loss, problems in achieving target freeness and pulp strength levels associated with the current dry blue-stained LPP chip supply. They have expressed significant concerns about their ability to utilize the longer-term wood chip supply comprising grey-stage chips. According to the limited information available from TMP pilot plant studies in the literature, there was a tendency towards reduced strength properties when refining wood that had been dried to moisture contents below FSP (Eriksen et al. 1981). There have been reports suggesting that chip presteaming and pretreatment with compression screw feeders or impressafiners can lead to improved remoistening of wood with low moisture content (Barbe et al. 1993; Parkinson et al. 1996) and that increasing the wood moisture content would improve the pulp properties (Eriksen et al. 1981; Hartler 1977; Barbe et al. 1993). In order to thoroughly evaluate these claims with respect to dry blue-stained and grey-stage wood, in advance of any capital spending programs that mechanical pulp mills may need to consider, a thorough laboratory assessment must be undertaken in order to test our industry's ability to effectively utilize this fibre supply, to develop appropriate pretreatment options and to retain market shares for BC mechanical pulps and papers.

1.2 Project objectives

The overall goal of this project is to determine the level of dry blue-stained wood chips and the level of grey-stage wood chips that can be economically utilized by BC mechanical pulp producers.

1.2.1 Target objectives

The target objectives are two-fold:

(1) to determine the maximum levels of dead, dry blue-stained and grey-stage lodgepole pine that can be economically utilized in BC mechanical pulp mills without detrimentally affecting processing and product quality; and

(2) to evaluate the economic feasibility of presteaming, chemical impregnation pretreatment and refiner operation options for dry blue-stained and grey-stage lodgepole pine wood chip utilization.

2 Material and Methods

2.1 Chip preparation and chip characterization

2.1.1 Green, early-grey and late-grey chips

Green, blue-stained early-grey (with a kill date of 2 years) and late-grey (kill date of 7 to 8 years) lodgepole pine (LPP) trees were harvested from sites with similar qualities in the interior of BC. The trees were transported to Paprican's Vancouver laboratory where selected bolts were debarked and then segregated by a portable sawmill to produce mature (over 40 years) sapwood sections. The mature sapwood sections of the green, early-grey and late-grey samples were chipped individually using a CM&E 10-knife disc chipper. Each of the three chip samples was screened on a Burnaby Machinery and Mill Equipment Ltd. two-deck laboratory chip classifier to remove oversize (>31 mm) and fine (<8 mm) material before their moisture content (MC) (wet basis) was determined. Chip packing density was determined by the method described by Hatton (Hatton 1979). Chip basic density was determined according to a modified PAPTAC Standard Method A.8P.

2.1.2 Chip blends and air-dried green and early-grey chips

Chip blends of 75/25, 50/50 and 25/75 [weight/weight, oven dried (o.d.) basis] green/late-grey LPP chips, and of 50/50 green/early-grey LPP chips were prepared from the corresponding amounts of the screened green and late-grey chips, and the screened green and early-grey chips, respectively. Each of the chip blends was well-mixed and its MC determined.

Portions of the screened green and early-grey chips were air-dried in a dark room at 23.8-24.1°C and 33.0-40.8% relative humidity over a period of 4-7 hours to ~17.0% MC by placing the chips evenly on a number of plastic mats, periodically monitoring the weights of a known amount of a small sample of the green and the early-grey chips, respectively.

2.2 Conventional TMP pulping using 12" (30.5-cm) Sunds Defibrator TMP 300 single-disc and 12" Sprout Waldron open-discharge laboratory refiners, fibre and sheet properties

The screened green, early-grey and late-grey chips; their blends; and the air-dried, screened green and early-grey chips were, respectively, subjected to first-stage refining in a 30.5-cm Sunds Defibrator TMP 300 single-disc laboratory refiner that incorporates a Labview PC system to control and monitor the refining variables. Various amounts of dilution H_2O were added to the refiner to give a constant, discharge pulp consistency of approximately 25% for the refining of various chips and chip blends with different

moisture contents. Pertinent refining conditions for the first-stage TMP pulping are given in Table 1.

Table 1. First-stage refining conditions using a 30.5 cm Sunds Defibrator TMP 300 single-disc laboratory refiner.

Plates	rotor, No. 3809 modified; Stator, No. 3804 modified
Preheater pressure	152 kPa
Refiner housing pressure	186 kPa
Presteaming time	10 min (atmospheric pressure)
Residence time	10 min
Pulp consistency	24 to 27% OD pulp (cyclone exit)
Prex compression ratio	3:1

The high freeness (~470-530 mL CSF) pulp from each of the first-stage refining runs was given one or more passes in a 30.5-cm Sprout Waldron open-discharge laboratory refiner equipped with type D2A507 plates to give TMP pulps with Canadian Standard Freeness (CSF) values between ~70-180 mL.

After latency removal, portions of each pulp were screened on a six-cut laboratory flat screen and the screen rejects determined. Bauer-McNett fibre classifications were carried out on the screened pulps. Fibre length of each of the TMP pulps was determined on a Fibre Quality Analyzer (FQA) instrument. Handsheets (60 g/m^2) were prepared with filtrate/white water recirculation to minimize the loss of fines and tested for density, physical properties, and optical properties using PAPTAC standard methods. Handsheet roughness was measured on the smooth side of the sheet in Sheffield units (SU).

2.3 TMP pulping under conventional and low intensity refining with and without pretreatment of the chips using 55.9-cm diameter Andritz 22-1CP single-disc refiner for the first-stage refining

The same green, early-grey and late-grey chips were used as in the conventional refining trials described in the previous section.

2.3.1 Low intensity refining without chip pretreatment

In the first stage, the chips were refined under the conditions listed in Table 2.

Table 2. Low intensity refining trials. First-stage refining conditions using 55.9-cm diameter Andritz 22-1CP single-disc refiner.

Plates	D17C002
Preheater pressure	250 kPa (gauge)
Refiner housing pressure	250 kPa (gauge)
Preheating time	100 sec.
Refiner rotational speed	1800 rpm
Target discharge consistency	30 %
Feed rate	2.02 – 2.56 kg/min
Specific energy	345 – 400 kWh/odt

For the early-grey and the late-grey chip samples, each first-stage pulp batch was split in two halves and refined in two additional stages using an atmospheric 91.44-cm diameter Bauer 400 double disc refiner at rotational speeds of 1200 rpm and 900 rpm, respectively. A rotational speed of 1200 rpm represents the standard refining intensity, while 900 rpm represents low refining intensity. The green chips were only refined at standard refining intensity. In the third-stage refining, the specific energy was increased stepwise to produce a refining curve. The target discharge consistency was 25%, the plate pattern was Bauer 36104 (NiHard) and the feed rate was 2.52 kg/min.

The pulps were disintegrated at about 90°C (latency removal) prior to testing. The first pulps were screened prior to testing using a Somerville screen with 0.15 mm slots, due to high rejects (shives) contents. For the rest of the samples, the shives contents were so low that screening was omitted. The samples from the first and second stage were only tested for Canadian Standard Freeness (CFS), Somerville screen rejects (shives content), Bauer McNett fractions and length weighted average fibre length. The fibre length was determined using the Fibre Quality Analyser (FQA). The samples from the third stage were tested for handsheet properties using PAPTAC standard procedures. The brightness was measured on a pile of 60g/m^2 handsheets.

2.3.2 Low intensity refining combined with chip pretreatment

Only the late-grey stage chips were used for these trials. The chips were steamed at 100°C under atmospheric conditions for 10 minutes prior to the water impregnation. An additional 4 minutes were needed to bring the temperature to 100°C. The solids content after steaming was 78%-80%. The water impregnation was done using an Andritz-Bauer model 15.24 cm MSD press-impregnator and expansion in hot water. The temperature after water impregnation was 68°C and the solids content was 28%. After impregnation, the chips were brought to the TMP pilot plant with no significant delay. The primary stage conditions are listed in Table 3. The sulphite impregnation was done using the same procedure, but with a chemical charge based on dry wood of 0.15% DTPA and 3% sodium sulphite at pH = 6 instead of pure water.

Table 3. Chip pre-treatment trials. First-stage refining conditions using a 55.9-cm Andritz 22-1CP single-disc refiner.

Plates	D17C002
Preheater pressure	250 kPa (gauge)
Refiner housing pressure	250 kPa (gauge)
Preheating time	180 sec.
Refiner rotational speed	1800 rpm
Target discharge consistency	30%
Feed rate	1.43 – 1.62 kg/min
Specific energy	367 – 502 kWh/odt

The second and third refining stages were done as described in the previous sections, using only the low level of rotational speed. The pulp testing was also done as described in the previous section.

2.4 Chip steaming and impregnation

2.4.1 Chip steaming and impregnation studies using patented Weyerhaeuser conditioned chip impregnation test apparatus

Unless otherwise specified, the weight of chips, W_{chips}, is that of the wet chips (i.e. with moisture in the chips). A sample (W_{chips} = ~100 g) of the screened green, early-grey, late-grey chips, or the air-dried, screened green or early-grey chips was presteamed in a laboratory bench-top autoclave (Brinkmann 2540M) at various steam pressures/temperatures for 30 and 55 minutes, respectively. The presteaming time shown throughout the report is the total time (i.e. time to pressure/temperature plus time at pressure/time) for which the chips were placed inside the autoclave; typically it took 11 to 15 and 16 to 17 minutes for the autoclave to reach the temperatures of ~123°C and 130°C, respectively. The presteaming temperatures were ~100, 123 and 130°C, respectively, which corresponded to presteaming pressures of ~101, 220 and 280 kPa (14.7, 32.0 and 40.7 psi). The weight of the presteamed chips, $W_{steamed\ chips}$, was recorded at the end of presteaming treatment.

The presteamed chips or the control (i.e. without steaming) chips (W_{chips} = ~100 g) were placed inside the covered wire mesh basket and immersed in DI H_2O of a known temperature or in an aqueous solution of known composition, conductivity, pH and temperature for 30 minutes in the Weyerhaeuser chip impregnation test apparatus (see Appendix 4). Prior to the lowering and immersing of the chip-containing basket into the DI H_2O or an aqueous solution, the weight of the DI H_2O (or an aqueous solution) and its

container was tared. Upon the lowering of the chip-containing basket, the weight of "basket + chips" was measured immediately and designated as $W_{\text{"basket + chips" at time zero}}$ and then measured continuously for 30 minutes at an interval of every second on the analytical balance and recorded in the computer. The weight measured/recorded at an impregnation time of t at room temperature (RT) (~23°C), designated as $W_{\text{"basket + chips" at time } t}$, decreased continuously because of the impregnation of water, or water and chemical, into the chips. At the end of the impregnation experiment, the chips were dried in an oven (set at 108°C) for a minimum of 17 hours and the oven-dried (o.d.) weight of the chips, $W_{o.d.\ chips}$, was measured. The moisture contents (wet basis) of the initial or control chips ($MC_{initial}$), the (pre)steamed chips ($MC_{steamed}$), and the chips (steamed or control chips) at an impregnation time of t at RT ($MC_{impregnation,\ t}$) were calculated as follows:

$$MC_{initial} = \frac{W_{chips} - W_{o.d.\ chips}}{W_{chips}} \times 100\%$$

$$MC_{steamed} = \frac{W_{steamed\ chips} - W_{o.d.\ chips}}{W_{steamed\ chips}} \times 100\%$$

$$MC_{impregnation,\ t} = \left(1 - \frac{W_{o.d.\ chips}}{W_{\text{"basket + chips" at time zero}} - W_{\text{"basket + chips" at time } t} + W_{steamed\ chips\ (or\ control\ chips)}}\right) \times 100\%$$

For impregnation experiments at 50, 70 and 80°C, control experiments were first done with ~100 g of plastic balls (instead of chips) being placed inside the basket and the amounts of vapour loss, $W_{\text{vapor loss}}$, over a period of 30 minutes at an interval of every second were recorded. The moisture contents of the chips (steamed or control chips) at an impregnation time of t and a temperature of T (T = 50, 70 or 80°C) ($MC_{impregnation,\ t,T}$) were calculated similarly to the calculation of $MC_{impregnation,\ t}$ except that "$W_{\text{"basket + chips" at time zero}} - W_{\text{"basket + chips" at time } t}$" was replaced with "$W_{\text{"basket + chips" at time zero}} - W_{\text{"basket + chips" at time } t} - W_{\text{vapor loss}}$".

For conciseness and easy file processing, only MC data from impregnation times of 0 (before impregnation), 1, 2, 4, 6, 8, 10 and 30 minutes, are presented/used in this report. All the data presented from Figure 26-31 are average values of three separate runs unless otherwise specified.

2.5 Environmental Scanning Electron Microscope (ESEM) - Energy Dispersive Spectroscopy (EDS) studies

2.5.1 Sample preparation

Wood blocks from the mature sapwood sections of the green and the late-grey LPP disks were prepared by hand using a chisel and razor blades. The blocks used in the experiments had final dimensions of 1.0 cm (radial) x 1.0 cm (tangential) x 2.0 cm (longitudinal). Each block was weighed prior to any treatment. The blocks, with and

without presteaming at 123°C for 55 minutes, were soaked in (unless otherwise specified) a 10 wt% of silver nitrate ($AgNO_3$) solution at room temperature for 5 or 10 minutes. The $AgNO_3$-soaked blocks were then removed from the solution. The liquid on the surface of each of the $AgNO_3$-soaked blocks was desorbed by a blotter before the weight of the block was recorded. Each of the blocks was then immersed in liquid nitrogen for 10 seconds, placed in a glass vial and stored in a freezer. Finally, each of the blocks was freeze-dried in a VirTis AdVantage Freeze Dryer before being placed inside a sealed vial and stored in a dessicator.

2.5.2 ESEM - EDS studies

Prior to ESEM - EDS studies, each of the freeze-dried, $AgNO_3$-soaked blocks was split along the tangential plane in the center to expose the interior, tangential, mid-line face. The split sample was placed on an SEM stub using two-sided tape and then placed in a FEI Quanta 400F field-emission ESEM equipped with an EDAX Genesis energy dispersive spectroscopy (EDS) system incorporating a 10 mm^2 thin window SiLi detector. Samples (without application of coating) were examined in low-vacuum (LV) mode at a chamber pressure of 1.0 Torr.

The intensity of Ag (the tracer element) at various locations along the longitudinal and the tangential mid-lines of the split sample (Figure 32) was measured by EDS. X-ray spectra were collected from an area of approximately 0.14 mm by 0.12 mm at each location, thus sampling a portion of a few contiguous fibres. Spectrum collection time was 100 seconds at an accelerating voltage of 20 kV. Working distance was approximately 10 mm for all samples.

3 Results and Discussion

3.1 Chip quality

The chip size distributions, the chip packing and chip basic densities, and the moisture content of the green, early-grey and late-grey LPP chips are shown in Table 4. The early- and late-grey samples had slightly higher fine chip content and lower accept chip content than the green sample. The chip packing densities of the three samples were similar while the basic density of the early-grey chips was the highest and that of the late-grey chips the lowest. As expected, both the early-grey and the late-grey chips had significantly lower moisture contents (~21% and 17%) than the green chips (~52%) (Table 4).

Table 4. Characteristics of the whole green, early-grey and late-grey LPP chips.

	Green	Early-Grey	Late-Grey
Chip classification			
45 mm Round Hole (overlarge), %	0.0	0.3	0.6
10 mm Slot (overthick), %	8.4	8.6	11.8
7 mm Round Hole (accept), %	88.3	85.0	83.9
3 mm Round Hole (pins), %	3.3	5.3	3.6
Pan (fines), %	0.0	0.7	0.2
Chip density and moisture content			
Chip Packing Density, kg/m^3	212	218	204
Chip Basic Density, kg/m^3	406	424	382
Moisture Content (wet basis), %	51.8 (51.4)[a]	20.9 (21.2)[a]	17.2 (16.9)[a]

[a]values on the screened chips

3.2 TMP pulping using 12-inch (30.5-cm) Sunds Defibrator TMP 300 single-disc and 12-inch Sprout Waldron open-discharge laboratory refiners

3.2.1 Energy consumption, fibre properties and strength

Data for the TMP pulping of the green, early-grey and late-grey chips, and fibre and handsheet properties of the TMP pulps are shown in Appendix 1. To facilitate data analysis and discussion, the raw data for selected properties were standardized by interpolation or extrapolation to 100 mL CSF and are shown in Table 5.

The specific refining energy needed for the TMP pulps to reach a given freeness in the range of 67-178 mL CSF is shown in Figure 1. These data and those shown in Table 5 indicate that energy requirements to a given freeness in TMP pulping of the early-grey (kill date 2 years) and of the green chips were similar. This result is consistent with our earlier findings that blue stain had no obvious effect on the specific refining energy requirement (Hu et al. 2006) and that up to 3-year beetle-infested LPP trees showed no well-defined relationship between energy consumption for TMP production and length of time since beetle infestation (Gee et al. 2004). The energy requirement for TMP pulping of the late-grey (kill date 6 to 7 years) chips was somewhat higher than that of the green or early-grey chips in the first set of trials (Table 5 and Figure 1). It was, however, slightly lower in the second set of trials using a different refiner (see Section 3.3). The reason for this discrepancy is not known at this time.

The long-fibre (R-48) fraction, fines fraction (P-200) and length weighted fibre length at 100 mL CSF were not very different among the green, early-grey and late grey TMP pulps (Table 5). During the second set of trials, where reduced energy was observed for the late-grey TMP, there were indications of reduced long fibre content (Figure 13 and Figure 14). The standardized data in Table 5 and plots of tensile strength of the TMP pulps vs. freeness values (Figure 2 and Figure 17) showed that the green and early-grey TMP pulp handsheets had similar tensile strength at the same freeness in spite of their different degree of, or time since, beetle infestation, and moisture content. The tensile index of late-grey TMP was slightly lower, in the order of 2-3 Nm/g at CSF=100 mL. These results are in accordance with anecdotal information from mechanical pulp mills using the current dry blue-stained LPP chips supply. Similar data had been obtained from TMP pulping of additional green, early-grey and late-grey samples (Dalpke et al. 2007).

Table 5. Selected properties of the green, early-grey and late-grey LPP TMP pulps at 100 mL CSF.

	Green	Early-Grey	Late-Grey
Specific energy (MJ/kg)	**9.9**	**9.7**	**11.3**
R-48 fraction (%)	**57.9**	**60.6**	**58.2**
Fines (P-200) (%)	**26.6**	**24.3**	**27.3**
Length weighted fibre length (mm)	**1.54**	**1.57**	**1.64**
Apparent sheet density (kg/m^3)	**364**	**350**	**346**
Tensile index (N·m/g)	**40**	**40**	**38**
Tear index ($mN·m^2/g$)	**7.8**	**7.8**	**8.5**
Sheffield roughness (SU)	**197**	**233**	**268**
Scattering coefficient (cm^2/g)	**564**	**531**	**624**
ISO brightness (%)	**57.0**	**50.6**	**51.2**
ISO opacity (%)	**95.2**	**97.0**	**97.7**

3.2.2 Optical properties, fibre consolidation and surface properties

The brightness values of the early-grey and late-grey TMP pulps were 6.4 and 5.8 ISO points lower than that of the green TMP pulp, while the opacity values of the early-grey and late-grey pulps were slightly higher than that of the green pulp (Table 5). The light scattering coefficient was significantly higher for the late-grey TMP. It is not clear if this was related to the moisture content or the infestation or due to natural variation in fibre properties. Alarmingly, handsheets made from the early-grey and late-grey TMP pulps had lower densities and higher roughness on the smooth side of the handsheets than those made from the green TMP pulp at any given freeness (Table 5, Figure 3 and Figure 4). The low sheet density and high handsheet surface roughness of the early-grey and late-grey TMP pulps could pose a significant problem for mills producing high-quality mechanical printing and writing papers, if bleaching of these pulps and calendering of the

sheets do not increase the sheet density and lower the surface roughness to a greater extent than bleaching and calendering of the green samples.

3.2.3 Maximum levels of dry early-grey and late-grey chips allowed

To determine the maximum levels of dry early-grey and late-grey chips that can be added to the green chips without significantly increasing the sheet handsheet roughness or lowering the density of the TMP pulps, or without significantly increasing the energy requirements for TMP pulping of the late-grey chips using the 30.5-cm Sunds Defibrator TMP 300 single-disc laboratory refiner (1st-stage refining) with preheater and refiner housing pressures of 152 and 186 kPa, chip blends of 75/25, 50/50 and 25/75 green/late-grey chips, and of 50/50 green/early-grey chips were used for TMP pulping. The roughness, density and energy requirement data (Appendices B and C) of the TMP pulps were compared with those of the pulps from green, late-grey and early-grey chips (Appendix 1).

In terms of energy requirements, it appeared that up to approximately 50% of the late-grey chips could be added to the green chips (Figure 5). Approximately 25% of the late-grey chips could be added to the green chips without significantly increasing the handsheet surface roughness (Figure 6) or decreasing the sheet density (Figure 7). It appeared that 50% of the early-grey chips could be added to the green chips without significantly increasing the handsheet surface roughness (Figure 8) but not without decreasing the sheet density (Figure 9).

3.2.4 Causes for the lower sheet density and higher handsheet surface roughness of the early-grey and late-grey TMP pulps

To understand the causes for the low sheet density and high handsheet surface roughness of the early-grey and late-grey TMP pulps, we air-dried our green and early-grey chips to moisture content the same as or close to that of our late-grey chips and subjected such air-dried chips to a TMP pulping and properties evaluation. The results (see data in Appendix 3) show that the low moisture content of dry late-grey and early-grey chips was not responsible for the higher energy requirement for the TMP pulping of the late-grey chips using the 30.5-cm Sunds Defibrator TMP 300 single-disc laboratory refiner (first-stage refining) with preheater and refiner housing pressures of 152 kPa and 186 kPa (Figure 10) or for the higher handsheet Sheffield roughness of the early-grey and late-grey TMP pulps (Figure 11). For example, the energy requirements for TMP pulping of the air-dried green chips (~17% MC) was practically the same as those for TMP pulping of the green chips (~52% MC), and lower than those for TMP pulping of the late-grey chips (~17% MC) (Figure 10). Other changes or the prolong (6-7 years) standing of the beetle-killed, dead trees at the dry state in the forest was likely responsible for the high handsheet roughness of the TMP pulp. These results demonstrate that one can't simply extrapolate the literature data/conclusions on dry wood mechanical pulping and apply them to pulping of the dry, beetle-infested wood. Much remains to be learnt regarding the behaviour of dry, late-grey LPP wood/chips.

The low moisture content of the early-grey and late-grey chips was, however, responsible for the low sheet density of the early-grey and late-grey TMP pulps (Figure 12).

3.3 TMP pulping under conventional and low refining intensities with and without pretreatment of the chips

3.3.1 Low intensity refining

Low intensity refining in the post-primary refining stages has previously been shown to help preserve the fibre length as the refining progresses (Miles and Omholt 2004). As shown in Figure 13-15, this was found also for the early-grey and particularly late-grey LPP. Reducing the refining intensity normally also leads to higher specific energy at a given CSF, which was also found in this case (Figure 16). The tensile index at a given CSF was not significantly affected by the refining intensity, as shown in Figure 17. The tear index at a given tensile index improved somewhat as expected due to the improved fibre length (see Figure 18). These results are in accordance with earlier findings. The complete set of results is shown in Appendix 4. The results indicate that reducing the refining intensity would improve the situation if the wood quality deteriorates to the point where preserving the pulp fibre length becomes a problem.

3.3.2 Low intensity refining combined with chip pretreatment

Two trials were done to explore options for improvement of the pulp strength from grey-stage wood if necessary through chip pretreatment. The conditions were chosen in such a way that an existing TMP mill that already has a compression screw installed should be able to adapt to them without major modifications. Only low intensity refining was used in these trials, in order to avoid compromising the strength improvements by fibre shortening in the post-primary refining stages.

As shown in Figure 19, the sulphite treatment at pH=6 gave a significant increase in the tensile index at a given CSF. This was expected due to the effect of sulphonation on the bonding ability of the fibres. The handsheet density also increased approximately 0.02 g/cm^3, as shown in Figure 20. The extent of these effects can be regulated by varying the sulphite charge, so the results shown here should be taken as illustrations of the direction in which this type of treatment will influence the pulp properties. The fibre length and the tear index were also improved by this treatment as shown in Figure 21 and 22.

The water impregnation, using an identical procedure to the sulphite pre-treatment except for the chemical charge, had only a marginal effect on the pulp properties. It may have produced a small increase in the tensile index at a given CSF, as shown in Figure 19. The screw press impregnation reduced the chip solids content to 28%, which is within the normal range for these kinds of trials.

Both treatments increased the specific energy at a given CSF, as shown in Figure 23. This may be partly due to an increase in the preheating time from 100 to 180 seconds in the case of the water impregnation. The sulphite pretreatment also led to a reduction of

the light scattering coefficient compared to untreated late-grey wood, as shown in Figure 24, and it improved the brightness somewhat, from about 53% to about 57% ISO. It is likely that it would cause a few percent yield loss and a corresponding increase in soluble organic material, but this was not measured during the trial. The complete set of results is shown in Appendix 5. Process modifications involving careful sulphite pre-treatment and treatment economics could be investigated further if loss of tensile strength due to beetle-killed wood becomes a greater problem in the future.

3.4 Chip steaming and impregnation studies

3.4.1 Reproducibility of the water-uptake data measured using Weyerhaeuser chip impregnation test apparatus during water soaking/impregnation of the early-grey chips

The Weyerhaeuser chip impregnation test apparatus (also referred to as chip fissure test device) (see Appendix 6) was developed and has been used for estimating kraft pulping yields based on the water absorption rates of oven-dried chips within 35 seconds of immersing the chips into the water (Levie et al. 1999; Levie and Marrs 2006). The apparatus employs a variant of "Archimedes Principle" which states that an object immersed in a fluid is buoyed up by a force equal to the weight of the displaced fluid. The main advantage of this apparatus is its ability to monitor the uptake/absorption of water into the oven-dried chips continuously and precisely over a period of 35 seconds, the time scale that has been used thus far.

We first modified the computer program of the Weyerhaeuser test apparatus to allow a continuous collection of data over a period of 30 minutes instead of 35 seconds, and then tested its ability to measure water absorption/impregnation into samples of the early-grey chips with moisture contents (wet basis) of 21.0%-22.2% instead of oven-dried chips. Data of the amounts of water absorbed/impregnated into (shown as moisture content of) the early-grey chips during soaking/impregnation of the chips with DI H_2O from three separate runs are shown in Figure 25. The moisture content of the early-grey chips increased rapidly over the first 4 minutes from ~21.6% to 33.0% gradually over the next 6 minutes to 35.4% and then slowly for the next 20 minutes to 37.5%. Excellent reproducibility was found from one run to another in spite of the heterogeneous nature of the chips.

3.4.2 Effect of presteaming temperature/pressure and time, composition, conductivity, temperature and pH of the impregnating/soaking solution, and its implications

After establishing the ability and reproducibility of the Weyerhaeuser test apparatus, we used it to evaluate the effect of presteaming of the early-grey chips on their subsequent water impregnation/uptake, and to study the effect of the composition, conductivity, temperature and pH of the impregnating solution. Both the presteaming temperature/pressure and the presteaming time were found to have a significant effect on

the water-impregnation/uptake of the early-grey chips during presteaming and subsequent water-impregnation (Figure 26). Presteaming of the chips at 100°C for 55 minutes (total time) and 123°C for 30 min increased the moisture contents by 1.0% and 2.1%, respectively. When such presteaming was followed by water impregnation at room temperature (RT), a synergistic increase in moisture content of the chips was observed. For example, after 8 minutes of water impregnation/soaking, additional moisture content increases of 2.3% and 2.7% (instead of 1.0% and 2.1%) were obtained for the presteamed chips over the chips without presteaming (Figure 26). Longer presteaming time (55 minutes) at 123°C or 130°C led to higher moisture content increases (5.8% or 6.4%) of the chips. When presteaming at 123°C or 130°C for 55 minutes was followed by water impregnation at RT, the moisture content increase was additive instead of synergistic (Figure 26).

Figure 27 shows the increase of moisture content of the early-grey chips (MC = 20.3%-23.6%) when soaked/impregnated at RT in $CaCl_2$ or NaCl solutions at various ionic strengths/conductivities. A conductivity of ~200 µS/cm appeared to be optimal for water impregnation of the chips and the divalent cation, Ca^{2+}, more beneficial in increasing the water uptake by the chips than the monovalent cation, Na^{+} (Figure 27).

The effects of soaking/impregnating water temperature, and that of soaking/impregnating water temperature and optimal conductivity or presteaming, on the moisture content of the early-grey chips (MC = 20.5%-23.9%) are shown in Figure 28 and Figure 29, respectively. The temperature of the soaking water had little effect on the DI water impregnation of the chips, particularly during the first 12 minutes of water impregnation (Figure 28), and little or negative effect on water impregnation when optimal conductivity of the impregnating solution was used or when the chips had been presteamed (Figure 29).

In addition, it was found that with the use of optimal conductivity and an impregnating time of 10 minutes or so, the moisture content of the chips could be increased, even at RT, to the same level as presteaming of the chips at high temperature (~123°C) for 55 minutes (a more costly approach) followed by soaking (typically achieved in the mills as washing) of the chips at a "non-optimal" conductivity (in this case, DI water) for 4 minutes (time used in some mills) (Figure 29). Thus, a more economical approach to raise the moisture content of the dry, early-grey LPP chips prior to TMP pulping is to control the conductivity of the chip-washing water and to use a chip washing time of 8 to 10 minutes instead of increasing the chip-washing water or presteaming temperature. Increasing the chip-washing or presteaming temperature, an approach that some mechanical pulp mills were considering using prior to our work, not only will be costly, but will also result in an additional load on the mill's effluent system. We have disclosed the findings discussed above to one of the participating mills in this CFS project; the mill is now exploring the possibilities of improving water impregnation of their dry, beetle-killed LPP chips through the use of a chip-washing time of 10 minutes or so and a control of the conductivity of their chip-washing water, instead of trying to raise the temperature of the chip-washing water.

In separate experiments it was found that the optimal pH of the soaking/impregnating water was ~6.0; increasing the pH to 8.0 or decreasing the pH to 4.5 resulted in a small or significant decrease, respectively, in the water impregnation of the early-grey chips.

3.4.3 Effect of the initial chip moisture content and other beetle-induced changes

Figure 30 shows data of the moisture content of the early-grey chips (MC = 21.4%), late-grey chips (MC = 17.0%), green chips (MC = 53.8%) and the air-dried green chips (MC = 16.9%) vs. DI water impregnating time at RT. Little H_2O was impregnated to the green chips after 30 minutes of impregnation; the moisture content changed from 53.8% to 55.5%. The late-grey chips had a slightly higher water uptake/impregnation rate than the early-grey chips in the first 10 minutes or so, and late-grey chips could reach moisture content similar to that of the early-grey chips.

Interestingly, the green chips air-dried to the moisture content similar to that of the late-grey chips showed a much lower water impregnation rate (Figure 30). Similar results were obtained when the water impregnation rate of the presteamed, air-dried green chips was compared to that of the presteamed, late-grey chips (Figure 31). The moisture content of the air-dried green chips could only reach, for example, 25.8% after 10 minutes of water impregnation, compared to a moisture content of 33.9% for the late-grey chips (Figure 30). The moisture content of the presteamed, air-dried green chips could only reach 34.6% after 10 minutes of water impregnation, compared to a moisture content of 40.5% for the presteamed late-grey chips (Figure 31). These results showed that the water impregnation ability of the dry, late-grey chips was drastically different from that of the green chips air-dried to the same starting moisture content, and that one can't simply extrapolate the literature data/conclusions on dry wood water-uptake/impregnation to water-uptake/impregnation of the dry, beetle-infested wood.

3.5 ESEM-EDS studies

To evaluate the differences in wood structure between the green and the grey-stage LPP at the tracheid level and to explore ways to monitor water penetration into the chips during water impregnation/soaking, we performed ESEM - EDS studies on the green and the late-grey blocks (1.0 cm x 1.0 cm x 2.0 cm radial, tangential and longitudinal dimensions) that had been soaked, or had been steamed and then soaked in 10% silver nitrate ($AgNO_3$) solution at room temperature for 5 or 10 minutes. The Ag was used as a tracer element for the movement of the water, and its intensity/amount inside and at the edges of the blocks was assessed by ESEM-EDS analysis.

Prior to ESEM-EDS analysis, each of the $AgNO_3$-soaked blocks was split along the tangential plane in the center to expose the interior, tangential, mid-line face. Figure 32 shows the points (LR1 - LR7 in the longitudinal direction, TB1 - TB4 in the tangential direction) on the exposed interior face at which the Ag intensities were determined. If there are no significant cracks/fissures on/inside the blocks and if the concentration of Ag

in the soaking solution is such that the amount of Ag that moves with water into the blocks is above the EDS Ag detection limit, it is expected that the Ag concentrations should decrease towards the center (LR4 point) in both the longitudinal and tangential directions. The Ag concentrations at LR2 and LR6 should also be higher than those at TB2 and TB3 because the movement of liquid (and thus $AgNO_3$) through lumens in the longitudinal direction is normally faster than that through the cell wall (in the tangential and radial directions). Table 6 shows the weight changes of the late-grey and the green blocks after presteaming and/or soaking in $AgNO_3$ solutions. Our preliminary data for Ag intensities measured by EDS at various points along the longitudinal and the tangential lines of the $AgNO_3$-soaked, split green and late-grey blocks are listed in Table 7 and Table 8, respectively.

The manually-determined weight changes of the blocks after soaking in $AgNO_3$ solutions (and without presteaming) were in agreement with our water-uptake data measured using Weyerhaeuser test apparatus. For example, soaking of the late-grey blocks in 0.03% $AgNO_3$ solution with the same conductivity (200 µS/cm) as the optimal value found in our previous soaking experiments on early-grey chips (Figure 27) produced a higher percentage weight gain than soaking of the late-grey block in 10% $AgNO_3$ with a much higher conductivity. Little liquid was taken up by the green blocks during soaking in $AgNO_3$ solution. Presteaming of the late-grey blocks appeared to inhibit the subsequent liquid uptake of the late-grey block in 10% $AgNO_3$ solution, which is different from our previous results on H_2O-uptake after presteaming and DI-H_2O soaking (Figure 26). Whether this was due to the large difference in the conductivity of the soaking solutions remains to be investigated.

Table 6. Weight changes of the late-grey and the green blocks after presteaming (123°C, 55 min) and/or soaking (in 10% AgNO3 solution).

	Initial (g)	Presteaming	After presteaming (g)	Soaking time (min)	After soaking (g)
Late-grey	0.623	no	-	5	1.306
	0.570	no	-	10	1.459
	0.584	yes	0.662	10	1.144
	0.562	no	-	10[a]	1.578
Green	2.002	no	-	5	2.073
	1.911	no	-	10	1.972
	1.997	yes	1.846	10	1.935

[a]0.03% $AgNO_3$ solution (conductivity = ~200 µS/cm) was used for soaking.

All the blocks that had been soaked in 10% $AgNO_3$ solution for 10 minutes showed decreasing Ag intensity towards the block center (LR4), except for the LR6 point of the late-grey block (LG, 10 minutes), and the LR3, TB2 and TB3 points of the green block (G, 10 minutes). Overall, Ag intensities at LR2 and LR6 for these blocks were also higher than those of TB2 and TB3 of the same blocks. The LR3, TB2 and TB3 values for the G, 10-minute block were slightly lower than or the same as the LR4 value for the same block, due likely to the scattering of data resulting from the values being near the EDS Ag detection limit. Overall, the results indicated that soaking of the green and the late-grey blocks with 10% $AgNO_3$ solution, followed by ESEM - EDS analysis of Ag intensity, could provide qualitative information on the movement of water within 10 minutes of water soaking/impregnation.

The green (MC = ~54%) blocks soaked in 10% $AgNO_3$ solution showed much lower absorption of Ag than the late-grey blocks (MC = ~17%) along the longitudinal (Table 7) or tangential lines (Table 8). This result is consistent with, and validates, our water-uptake results obtained using Weyerhaeuser test apparatus which showed that little H_2O was taken up by the green chips even after 30 minutes of impregnation (Figure 30).

The late-grey block soaked in 10% $AgNO_3$ solution for 5 minutes showed non-uniform Ag distribution. This could be due to the fissures in the block that were evident during ESEM examination. An ESEM micrograph of this sample shows large fissures that extend to the center of the block (Figure 33). No such fissures were found in the green block soaked in 10% $AgNO_3$ solution for 5 min (Figure 34), and this sample showed the expected decrease of Ag intensity towards the center (LR4) of the sample. Future work on the monitoring of water movement into the grey-stage blocks/chips by ESEM - EDS analysis of Ag (the tracer element) should consider selection of samples without fissures. This may be difficult to achieve with dry wood samples.

Table 7. The Ag intensity at various longitudinal positions of the split green and late-grey wood blocks, respectively, after the blocks were soaked in 10% AgNO3 solution for 10 or 5 minutes without or "with steaming at 123oC for 55 minutes" (steamed) prior to soaking.

Sample, soaking time	Left edge			Centre			Right edge
	LR1	LR2	LR3	LR4	LR5	LR6	LR7
Late-grey (LG), 10 min	65.0	53.2	28.6	18.5	63.7	96.6	71.7
Green (G), 10 min	7.2	1.3	0.6	1.2	1.5	2.8	8.2
LG, 5 min	58.2	54.2	59.6	53.9	67.2	36.7	45.0
G, 5 min	8.8	3.1	2.2	0.3	1.2	1.6	9.6
Steamed LG, 10 min	56.7	31.5	1.1	0.8	1.4	21.2	66.2
Steamed G, 10 min	12.5	4.2	2.2	0.0	0.2	1.2	4.9
LG, 10 min[a]	0.5	0.7	0.4	0.6	0.5	1.1	1.4

[a]0.03% $AgNO_3$ solution (conductivity = ~200 µS/cm) was used for soaking.

Table 8. The Ag intensity at various tangential mid-line positions of the split green and late-grey wood blocks, respectively, after the blocks were soaked in 10% AgNO3 solution for 10 or five minutes without or "with steaming at 123°C for 55 minutes" (steamed) prior to soaking.

Sample, soaking time	Top edge		Centre		Bottom edge
	TB1	TB2	LR4	TB3	TB4
Late-grey (LG), 10 min	66.0	24.8	18.5	41.1	56.2
Green (G), 10 min	5.5	1.2	1.2	1.0	5.0
LG, 5 min	24.8	0.8	53.9	0.8	4.4
G, 5 min	9.9	1.6	0.3	0.6	4.6
Steamed LG, 10 min	23.2	1.9	0.8	18.5	40.4
Steamed G, 10 min	7.8	1.2	0.0	1.6	8.6
LG, 10 min[a]	1.0	1.0	0.6	0.6	1.2

[a]**0.03% $AgNO_3$ solution (conductivity = ~200 µS/cm) was used for soaking.**

Analysis of a late-grey block that had been soaked in 0.03% $AgNO_3$ solution with the same conductivity (200 µS/cm) as the optimal value found in our previous soaking experiments on early-grey chips (Figure 27) showed there was a small amount of Ag all through the sample. Although the conductivity was optimal for water penetration/uptake as confirmed by the higher percentage weight gain after soaking (Table 6), the concentration of Ag was so low in the soaking solution that the amount of Ag that moved with the water was near the EDS Ag detection limit.

Presteaming of the late-grey block appeared to make it difficult for the Ag ion to move towards the center of the blocks. This was consistent with our findings that presteaming inhibited the subsequent liquid uptake (thus fewer Ag ions) of the block in 10% $AgNO_3$ solution (Table 6). There are obvious challenges to including $AgNO_3$ in presteaming (distillation effect) and so it is unclear at this time how tracer compounds might be incorporated in presteaming so that EDS results from trials with and without steaming can be directly compared (quantitatively rather than qualitatively). Further studies are needed.

A close-up ESEM image of one of the late-grey samples showed the presence of fungal hyphae in the lumen of a fibre (Figure 35). The presence of fungal hyphae (obviously including but not limited to blue-stain fungi) was observed in all the late-grey samples. There was also some indication of incipient decay in the late-grey blocks (Figure 36). The lighter areas seen in the block may be due to decay. This image can be directly compared with Figure 32 which shows a green sample. As with fissures, decay in samples is difficult to avoid, particularly with samples where there has been a long time since death.

4 Conclusions

The energy requirements for TMP pulping of both the dry, early-grey (kill date 2 years) chips and the dry, late-grey (kill date 6-7 years) chips were similar to those for TMP pulping of the green chips when all the results were taken into consideration. The early-grey and late-grey TMP pulps had lower sheet density and ISO brightness, and higher handsheet surface roughness than the green TMP pulps. No significant difference in long-fibre and fines contents, fibre-length or tensile strength was observed between the green and the early-grey TMP pulps at the same freeness, while indications of slightly lower tensile index were found for the late-grey TMP. The late-grey TMP also showed a higher light scattering coefficient at a given CSF.

The higher handsheet surface roughness of the late-grey and early-grey TMP pulps was not due to the low moisture content of the late-grey and early-grey chips, but likely due to other changes. The lower sheet density of the early-grey and late-grey TMP pulps was, however, due to the low moisture content of the early-grey and late-grey chips. The maximum level of the dry, late-grey chips that could be added to the green chips without significantly increasing the handsheet surface roughness or lowering the sheet density was 25%.

Low-intensity refining in the post-primary stages of the early-grey and the late-grey TMP improved the fibre length, but it did not affect the tensile index at a given CSF significantly. Chip presteaming and water impregnation had a marginal effect on the pulp properties of the late-grey TMP. Sodium sulfite pretreatment at pH=6 coupled with low-intensity refining of the late-grey chips gave, however, a positive effect on the tensile index at a given CSF. Process modifications involving careful sulphite pretreatment could be investigated further if loss of tensile strength due to beetle-killed wood becomes a greater problem in the future. Reducing the refining intensity would represent a possible solution if maintaining the fibre length becomes a critical issue. Both these modifications led to somewhat increased refining energy consumption at a given CSF.

Increase of moisture content/water impregnation of the early- and late-grey LPP chips could be achieved by increasing chip presteaming temperature/pressure and/or time, by increasing the water impregnation time from, for example, 4 minutes to approximately 10 minutes, or by controlling the conductivity and pH of the impregnating water at approximately 200 µS/cm and ~6.0 µS/cm, respectively. The temperature of the chip

impregnation water had little effect on the water impregnation of the chips, particularly if the chip impregnation/soaking time was ≤10 minutes. The most economical pretreatment approach to increasing the moisture content of the dry, grey-stage LPP chips prior to TMP pulping appeared to be a presteaming at moderate temperature/pressure, followed by 8 to 10 minutes of chip washing with a proper control of the conductivity and pH of the chip-washing water.

Dry, late-grey LPP chips behaved very differently during TMP pulping or water/chemical impregnation than green LPP chips air-dried to the same starting moisture content. One can't simply extrapolate the data/conclusions in dry wood TMP pulping or water/chemical impregnation and apply them to pulping or water/chemical impregnation of the dry, beetle-infested wood.

Soaking of the green and the late-grey blocks in 10% $AgNO_3$ solution, followed by ESEM-EDS analysis of Ag intensity, could provide qualitative information on the movement of water within 10 minutes of water soaking/impregnation. ESEM-EDS results on the green and late-grey blocks are consistent with the water-uptake results obtained using Weyerhaeuser test apparatus.

5 Acknowledgements

This project was funded by the Government of Canada through the Mountain Pine Beetle Initiative, a program administered by Natural Resources Canada, Canadian Forest Service. Publication does not necessarily signify that the contents of this report reflect the views or policies of Natural Resources Canada – Canadian Forest Service. We would like to thank Dr. Benjamin E. Levie of Weyerhaeuser Company at Tacoma, Washington for lending us the Weyerhaeuser chip impregnation test apparatus; Dr. Barbara Dalpke of Paprican – Vancouver laboratory for providing us with the tree samples and data on the chip qualities and for reviewing this report; Derek Dranfield and Daniel Gilbert of Paprican – Pointe-Claire laboratory for their contribution in carrying out the pilot trials; and Gail Sherson and Dr. Paul Bicho of Paprican – Vancouver laboratory and Dr. Zhirun Yuan of Paprican – Pointe-Claire laboratory for helpful discussions.

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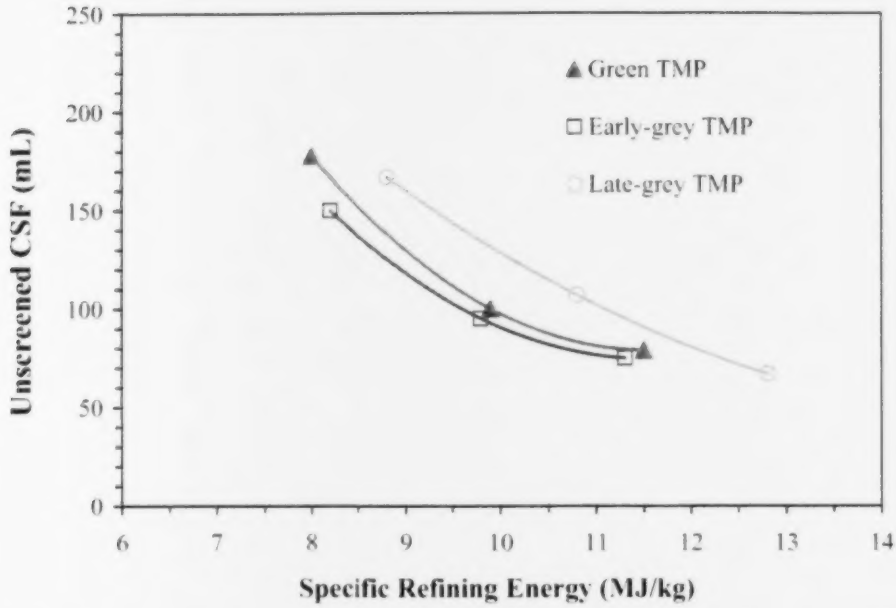


Figure 1. Energy requirements for TMP pulping of the green, early-grey and late-grey LPP chips.

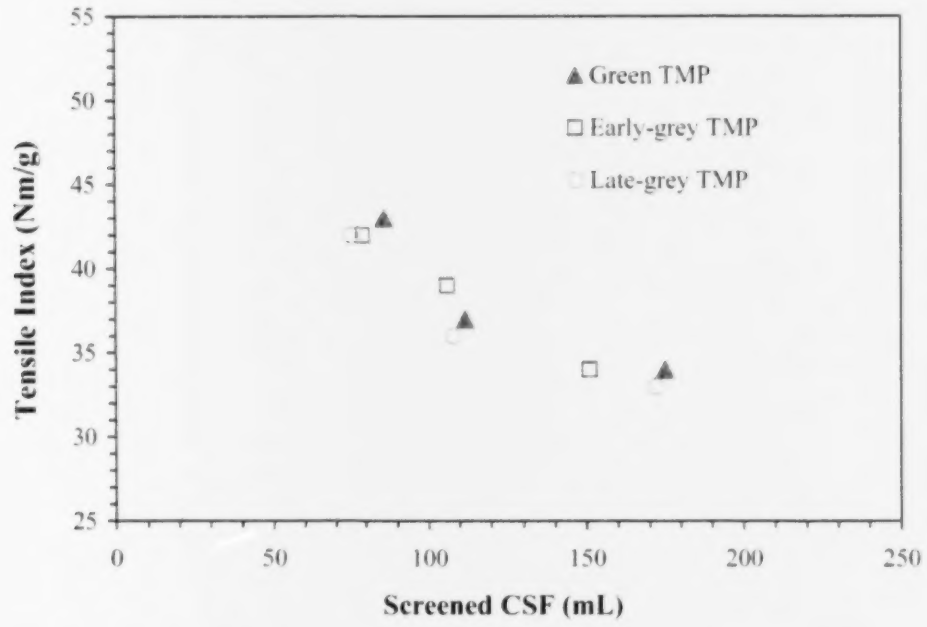


Figure 2. Tensile strength of the green, early-grey and late-grey TMP pulps.

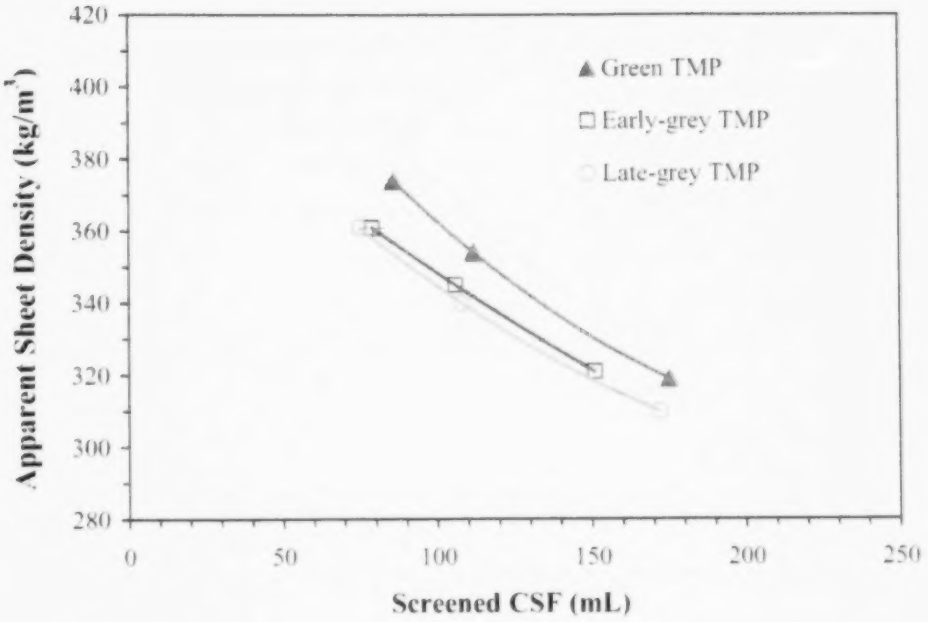


Figure 3. Apparent sheet density of the green, early-grey and late-grey TMP pulps.

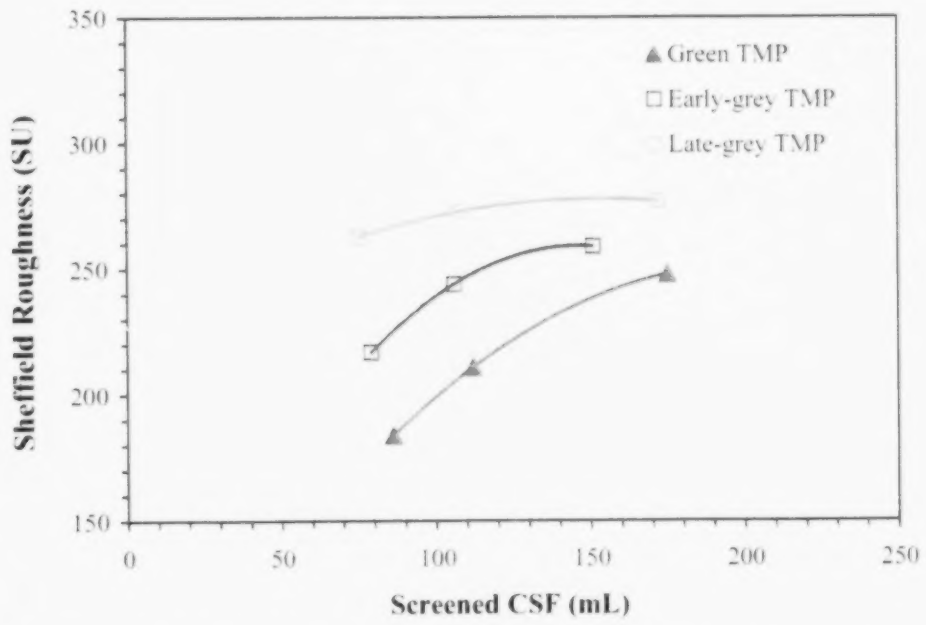


Figure 4. Sheffield roughness of the green, early-grey and late-grey TMP pulps.

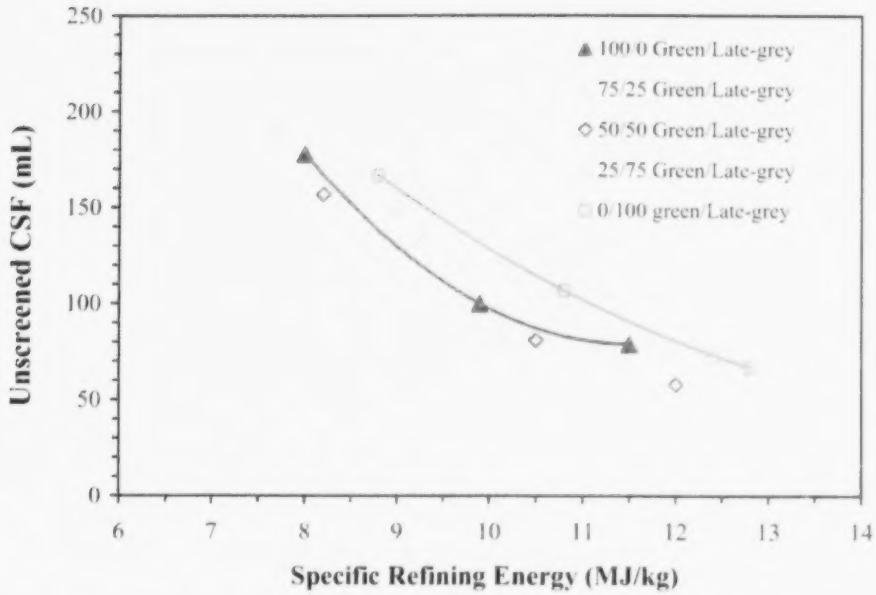


Figure 5. Energy requirement for TMP pulping of blends of the green and late-grey chips.

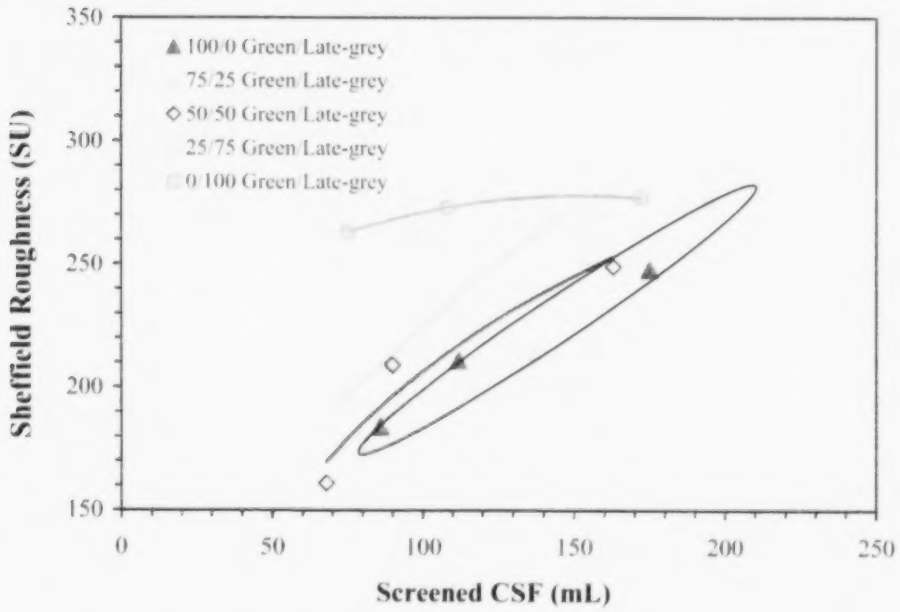


Figure 6. Sheffield roughness of the TMP pulps made from blends of green/late-grey chips.

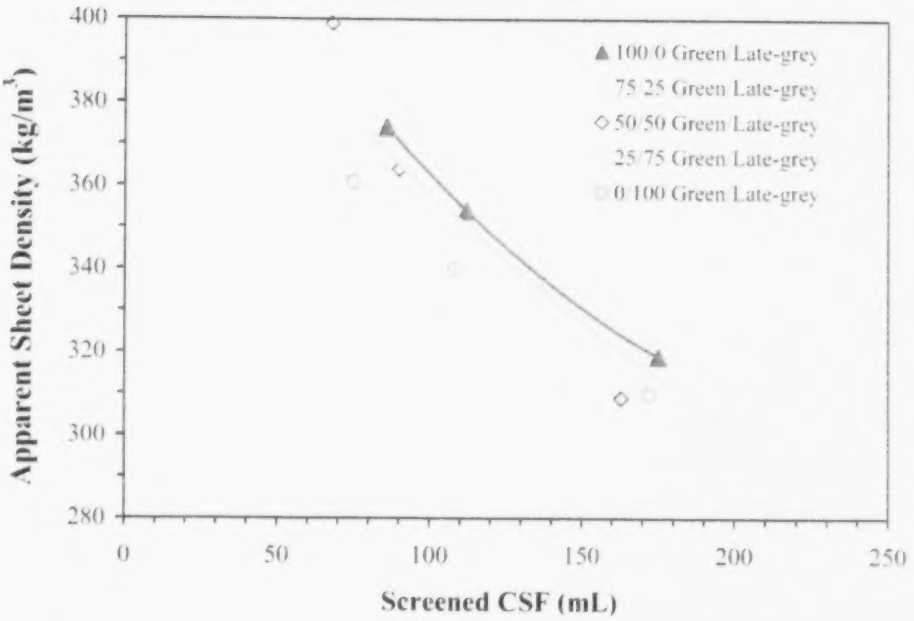


Figure 7. Apparent sheet density of the TMP pulps made from blends of green/late-grey chips.

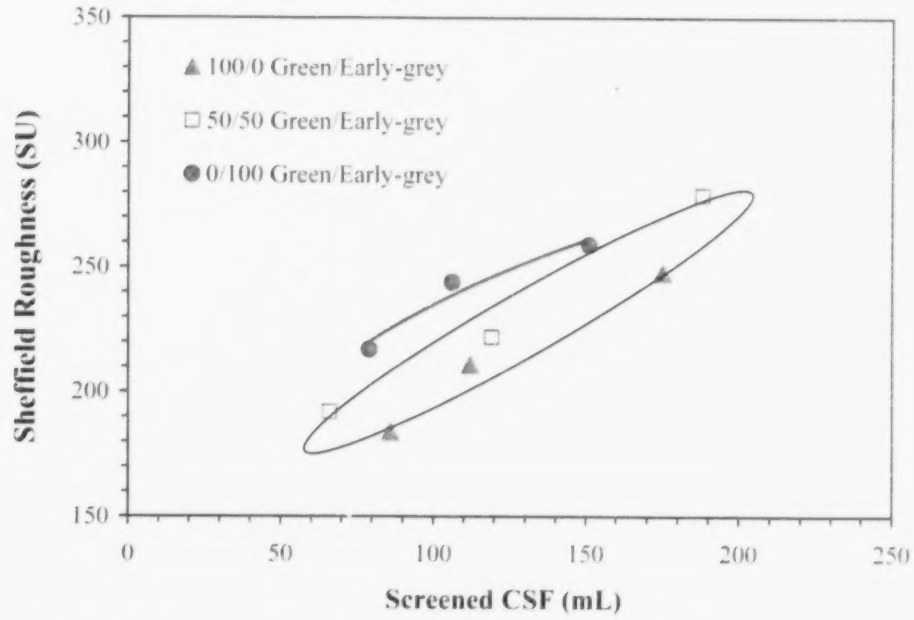


Figure 8. Sheffield roughness of the TMP pulps made from blends of green/early-grey chips.

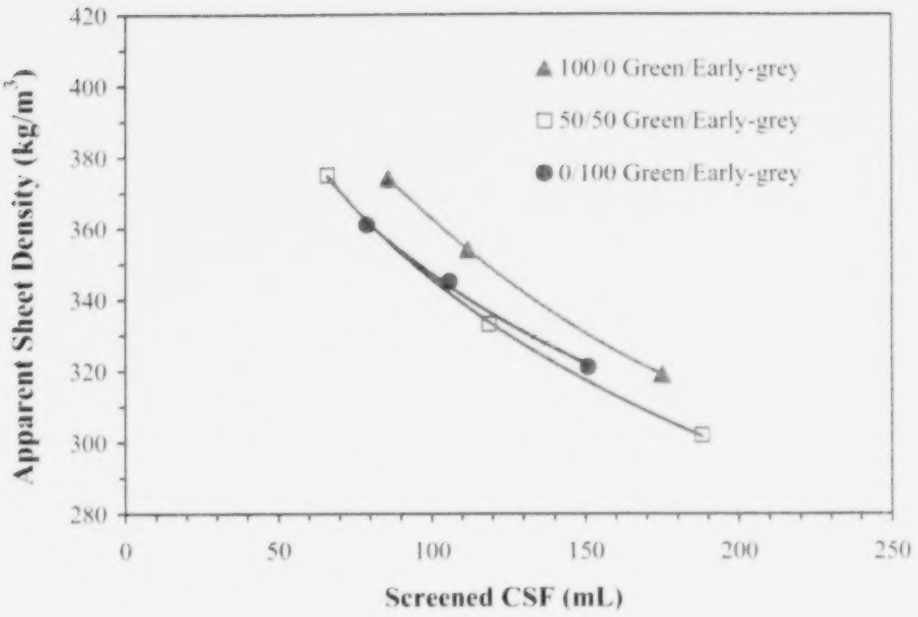


Figure 9. Apparent sheet density of the TMP pulps made from blends of green/early-grey chips.

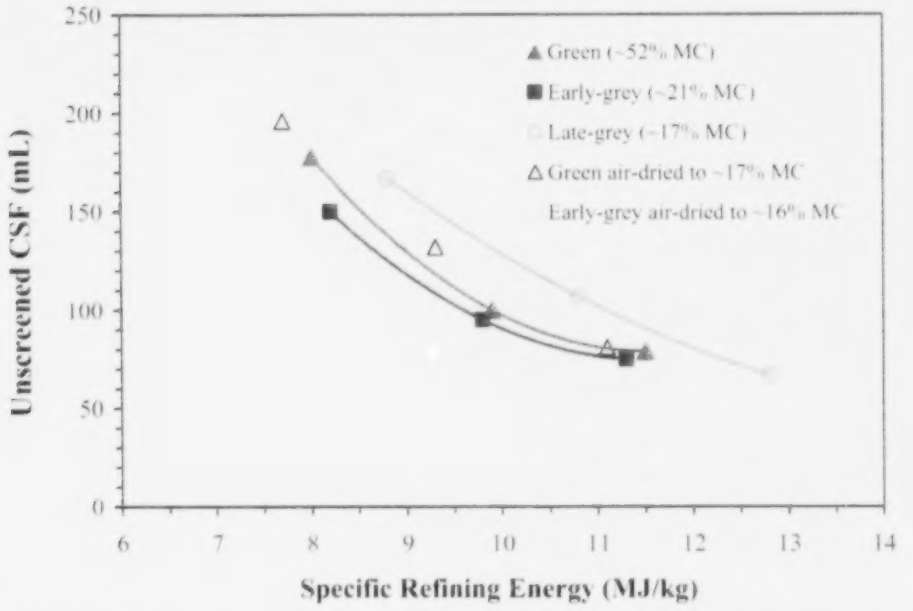


Figure 10. Energy requirement for TMP pulping of the green, early-grey and late-grey chips, and of the air-dried green and early-grey chips.

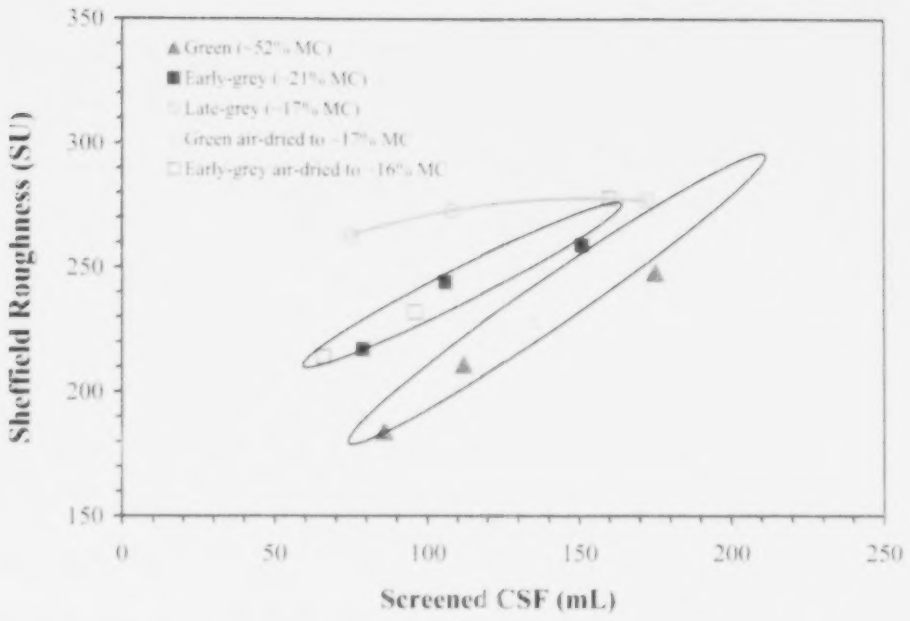


Figure 11. Sheffield roughness of the TMP pulps made from the green, early-grey and late-grey chips, and from the air-dried green and early-grey chips.

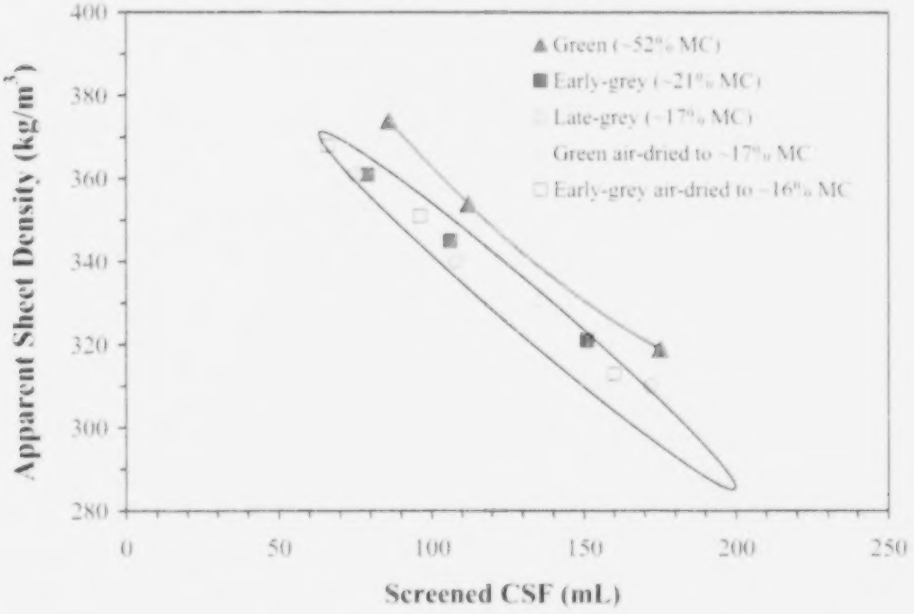


Figure 12. Apparent sheet density of the TMP pulps made from the green, early-grey and late-grey chips, and from the air-dried green and early-grey chips.

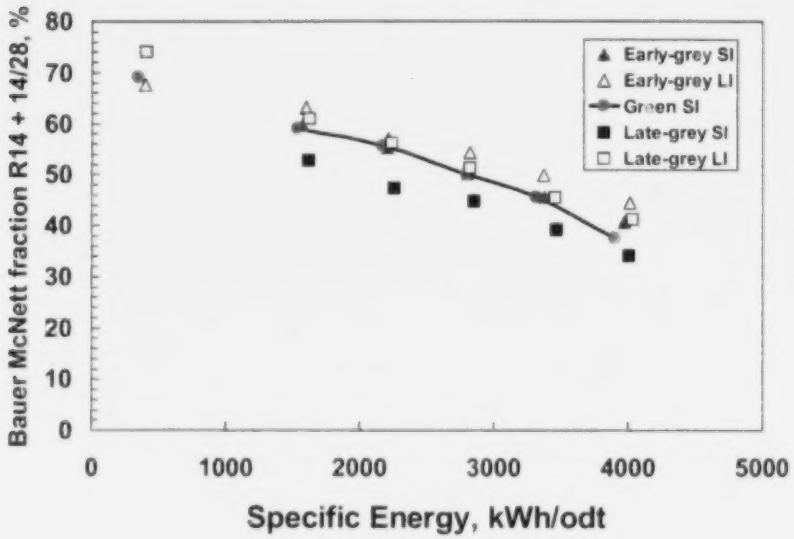


Figure 13. The effect of low intensity refining in the post-primary stages on the Bauer McNett fraction R14 + 14/28. SI=standard intensity, LI=low intensity.

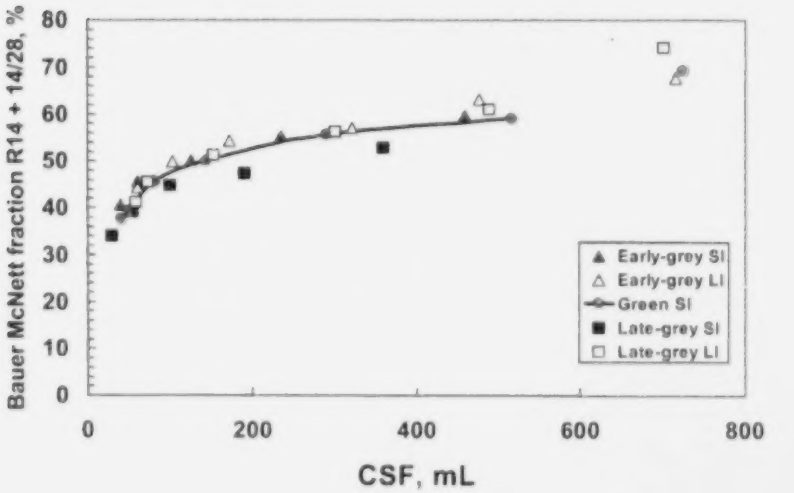


Figure 14. The effect of low intensity refining in the post-primary stages on the Bauer McNett fractions R14 + 14/28 at a given Canadian Standard Freeness. SI=standard intensity, LI=low intensity.

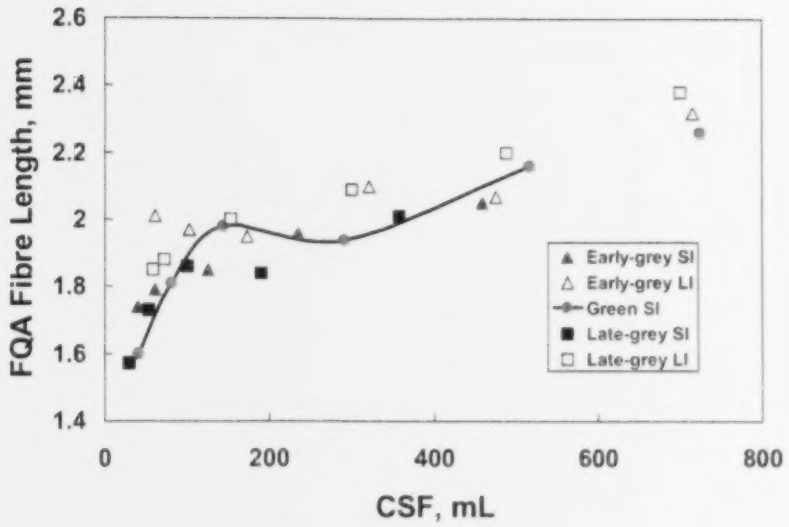


Figure 15. The effect of low intensity refining in the post-primary stages on the length weighted average fibre length at a given Canadian Standard Freeness. SI=standard intensity, LI=low intensity.

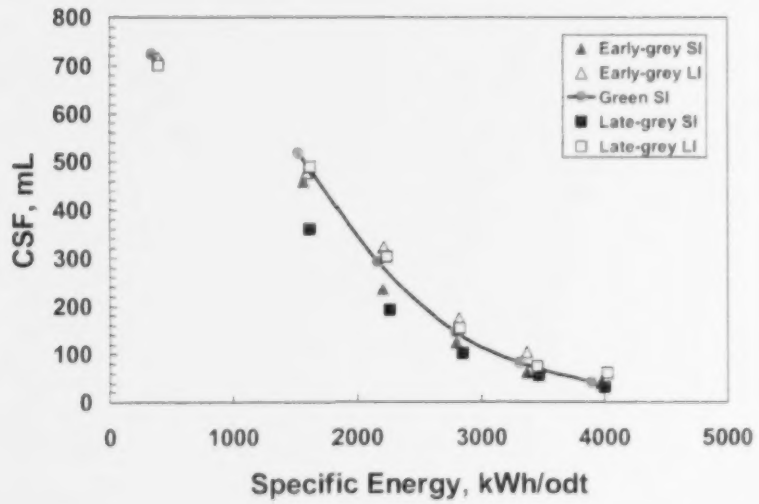


Figure 16. The effect of low intensity refining in the post-primary stages on the Canadian Standard Freeness at a given specific energy. SI=standard intensity, LI=low intensity.

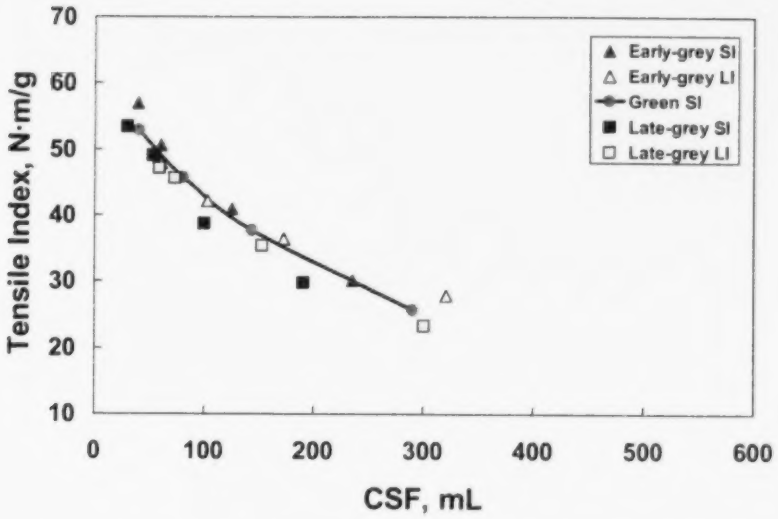


Figure 17. The effect of low intensity refining in the post-primary stages on the tensile index at a given Canadian Standard Freeness. SI=standard intensity, LI=low intensity.

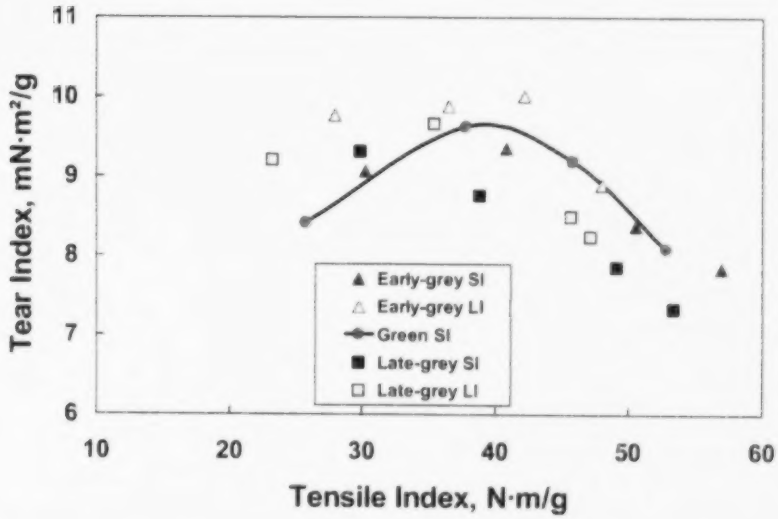


Figure 18. The effect of low intensity refining in the post-primary stages on the tear index at a given tensile index. SI=standard intensity, LI=low intensity.

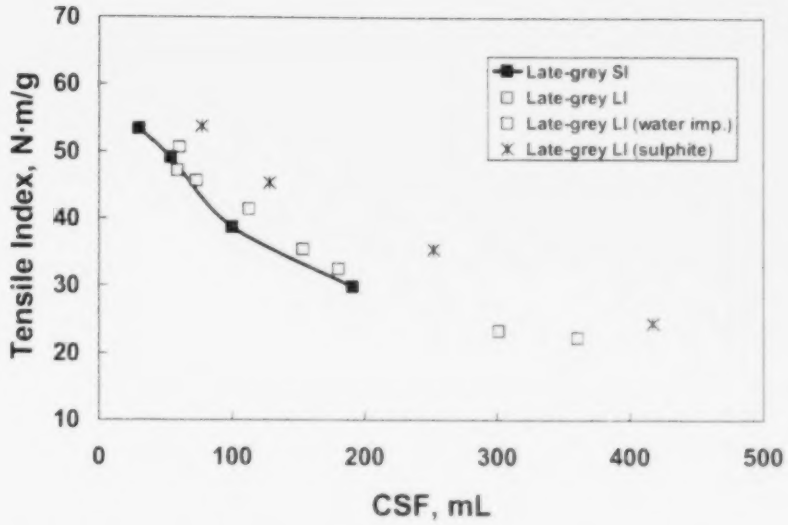


Figure 19. Tensile index at a given CSF for pretreated chips. SI=standard intensity, LI=low intensity.

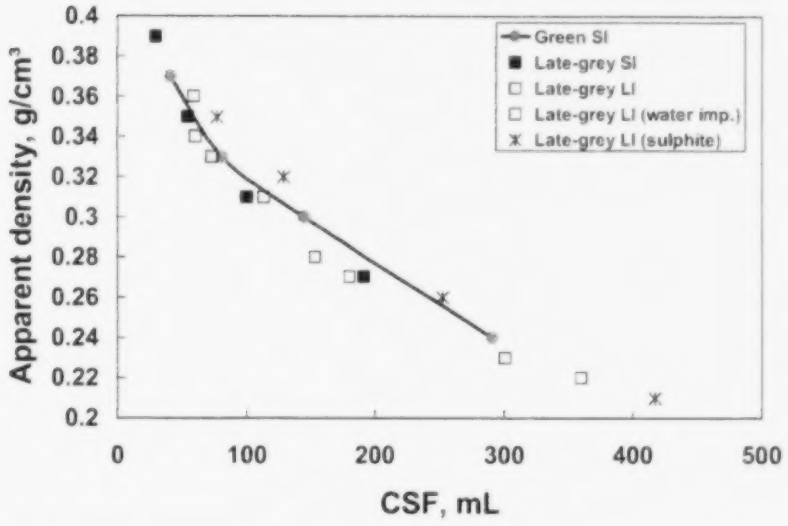


Figure 20. Handsheet density at a given CSF for pretreated chips. SI=standard intensity, LI=low intensity.

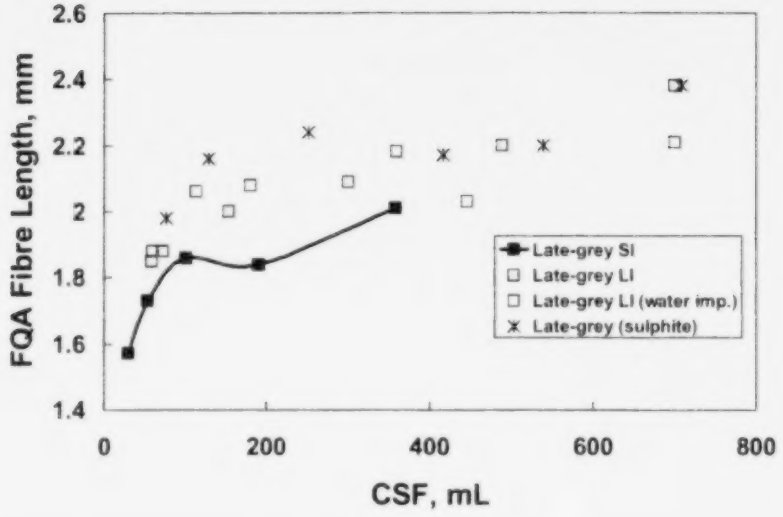


Figure 21. Fibre length at a given CSF for pretreated chips. SI=standard intensity, LI=low intensity.

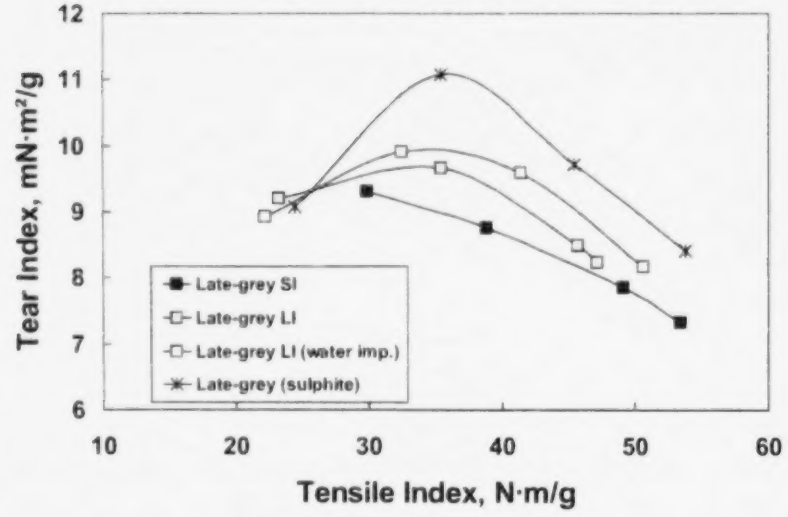


Figure 22. Tear index at a given tensile index for pretreated chips. SI=standard intensity, LI=low intensity.

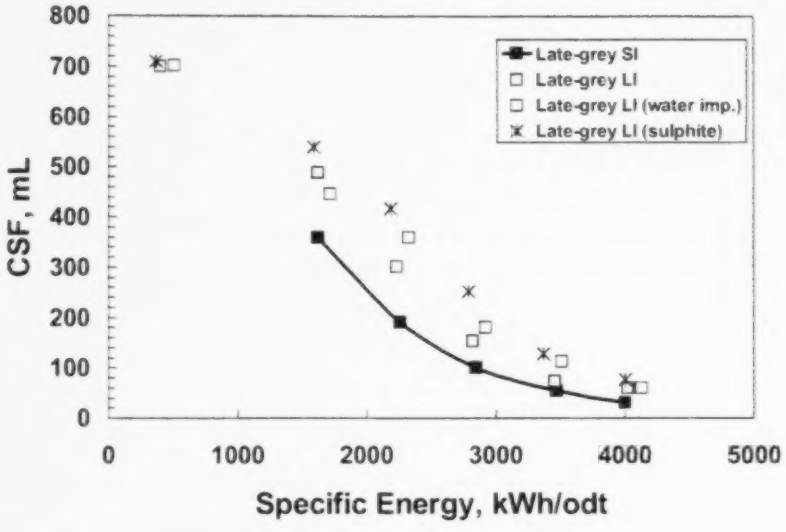


Figure 23. CSF versus specific energy for pretreated chips. SI=standard intensity, LI=low intensity.

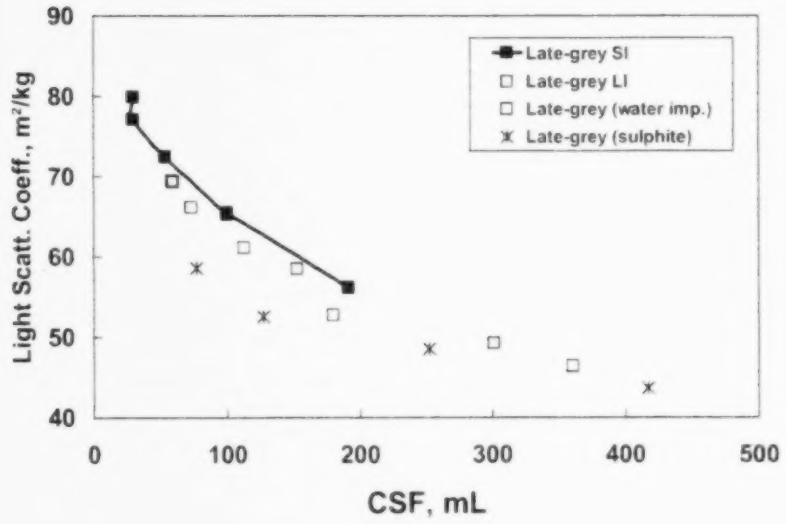


Figure 24. Light scattering for a given CSF for pretreated chips. SI=standard intensity, LI=low intensity.

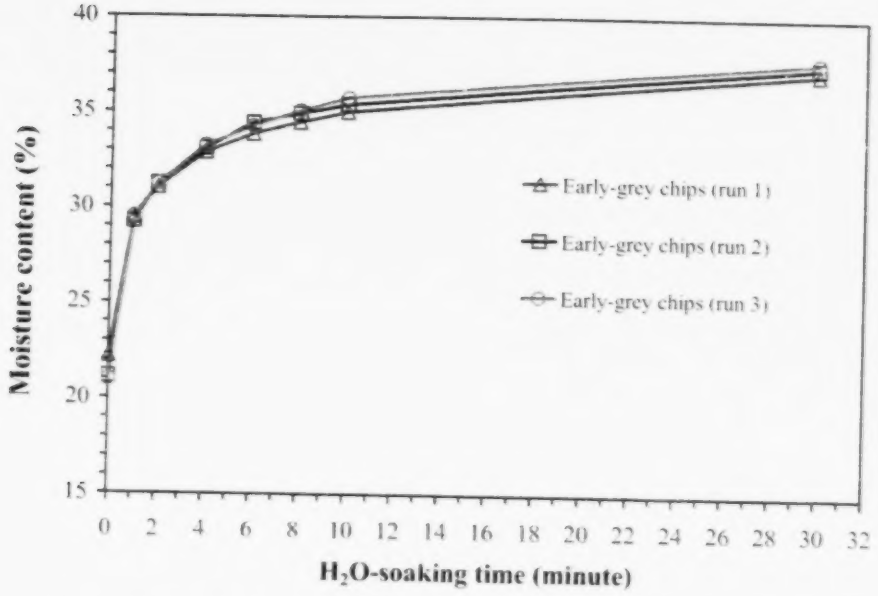


Figure 25. Moisture content (%) of the early-grey chips vs. DI water soaking/impregnation time at room temperature.

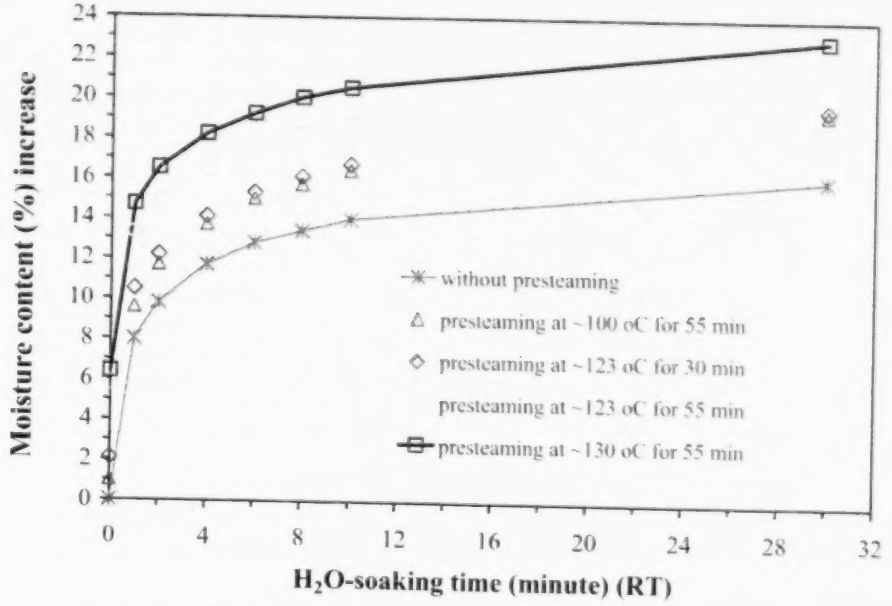


Figure 26. Increase in moisture content of the early-grey chips during room temperature DI water soaking/impregnation without or with presteaming.

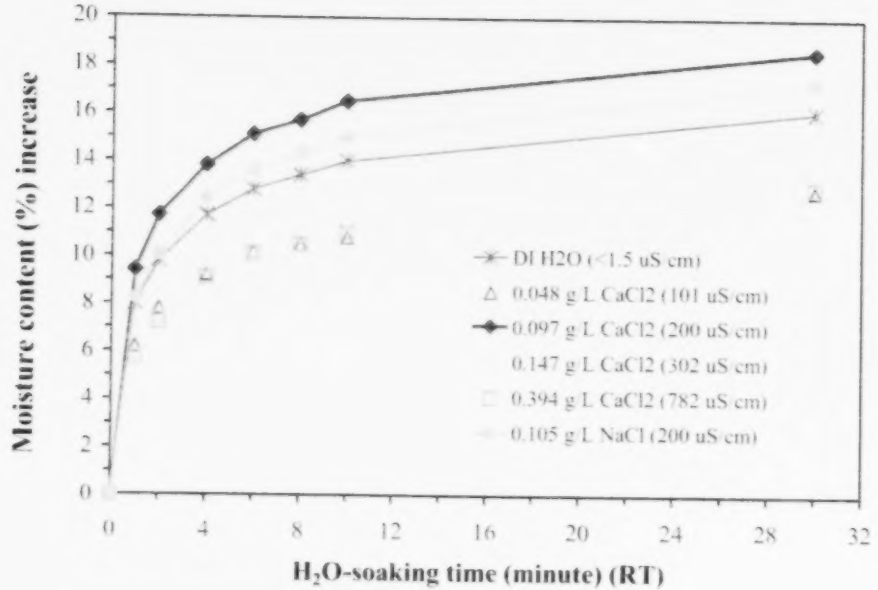

Moisture content (%) increase
H2O-soaking time (minute) (RT)
DI H2O (<1.5 uS/cm)
0.048 g/L CaCl2 (101 uS/cm)
0.097 g/L CaCl2 (200 uS/cm)
0.147 g/L CaCl2 (302 uS/cm)
0.394 g/L CaCl2 (782 uS/cm)
0.105 g/L NaCl (200 uS/cm)

Figure 27. Effect of the conductivity of, and the cation of the salt added to the soaking/impregnating water on water impregnation of the early-grey chips at RT; data for 0.048, 0.147 and 0.394 g/L CaCl2 experiments were from a single run.

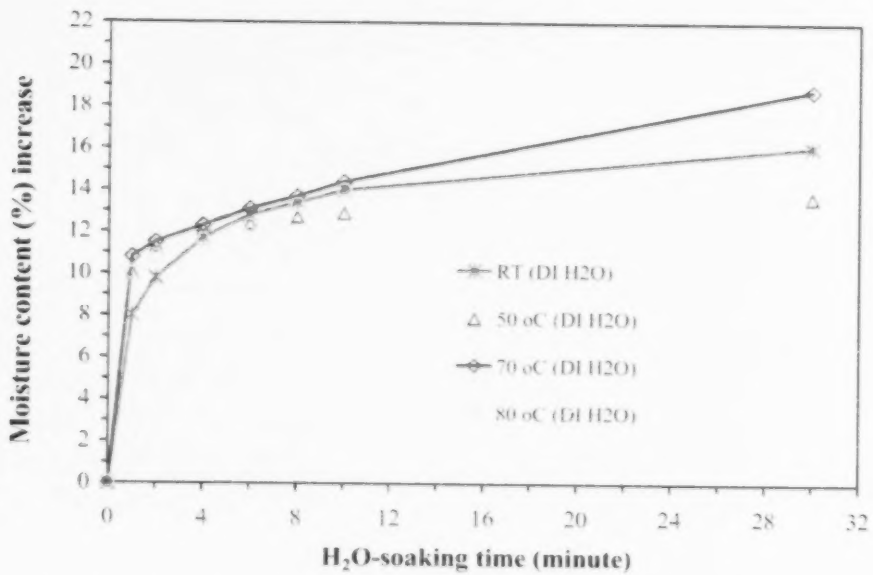


Figure 28. Effect of the soaking/impregnating water temperature on DI water impregnation of the early-grey chips.

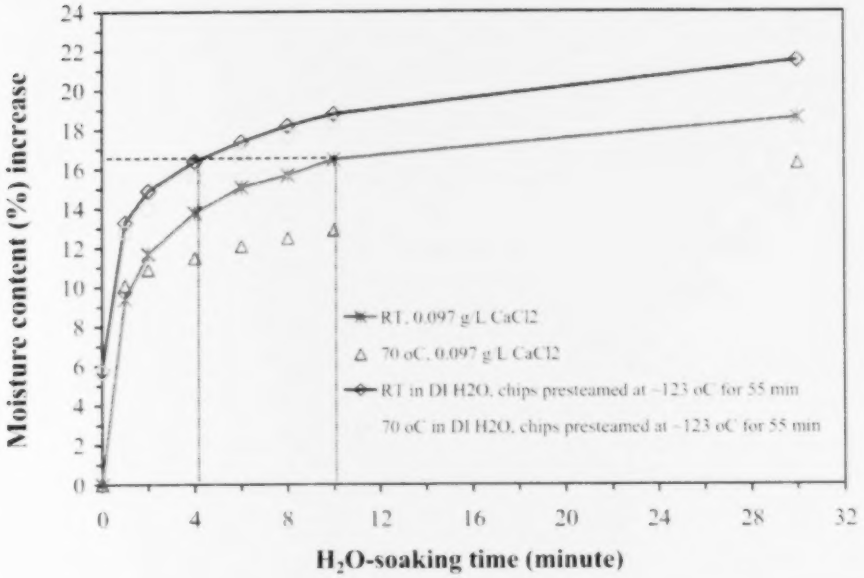

Moisture content (%) increase
H2O-soaking time (minute)
RT, 0.097 g/L CaCl2
70 oC, 0.097 g/L CaCl2
RT in DI H2O, chips presteamed at ~123 oC for 55 min
70 oC in DI H2O, chips presteamed at ~123 oC for 55 min

Figure 29. Combined effect of the temperature and optimal conductivity of the soaking water or chip presteaming on water impregnation of the early-grey chips; data for the "70°C in DI H2O, chips presteamed at 123°C for 55 min" experiment were average values of two runs.

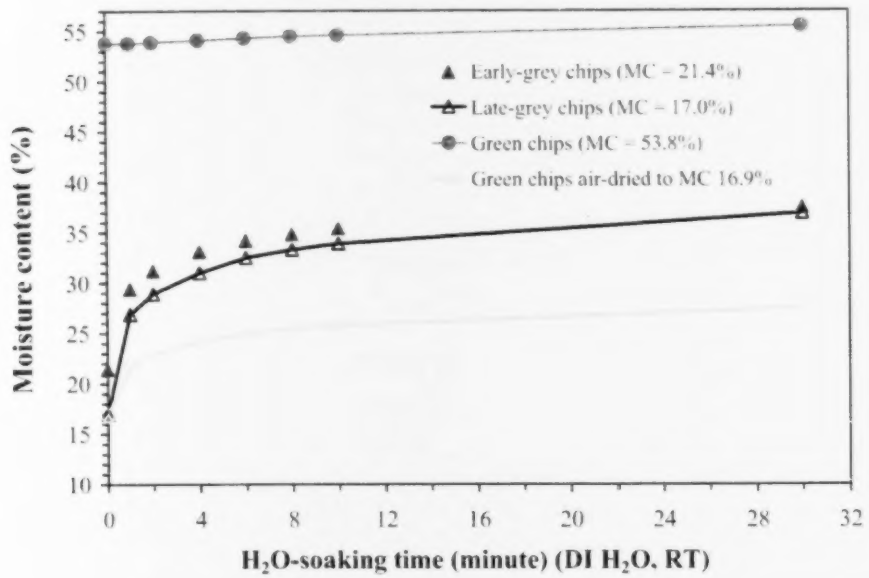

Moisture content (%)
H2O-soaking time (minute) (DI H2O, RT)
Early-grey chips (MC = 21.4%)
Late-grey chips (MC = 17.0%)
Green chips (MC = 53.8%)
Green chips air-dried to MC 16.9%

Figure 30. Moisture content of the early-grey, late-grey, green and air-dried green chips vs. DI water impregnating time at RT.

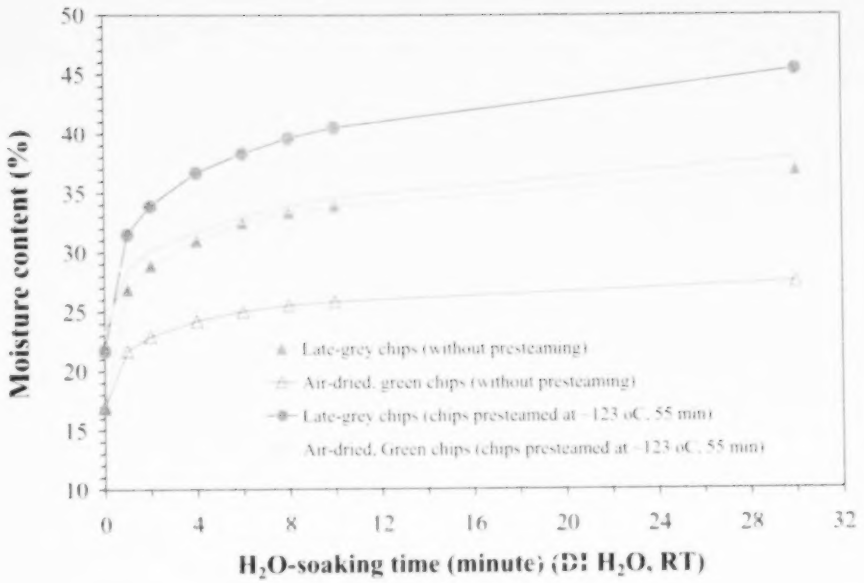


Figure 31. Moisture content of the late-grey chips and the air-dried, green chips without or with chip presteaming vs. DI water impregnating time at RT; data for the "air-dried, green chips (chips presteamed at ~123°C for 55 min)" were average values from two runs.

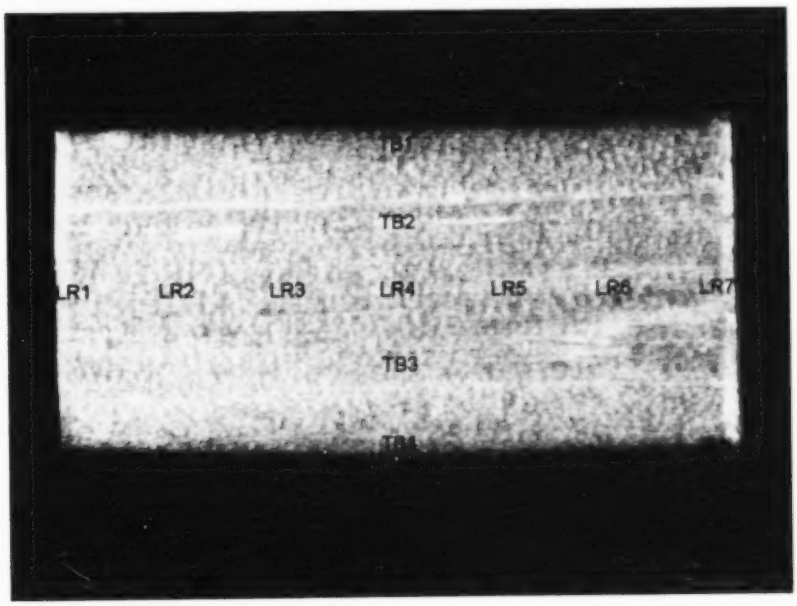


Figure 32. Locations on the exposed interior face of the $AgNO_3$-soaked block for Ag analyses by ESEM-EDS.

Figure 31. Moisture content of the kiln-grey chips and the air-dried green chips without or with chip presteaming vs. DI water impregnating time at RT; data for the "air-dried green chips/chips presteamed at 123 °C for 55 min" were average values from two runs.

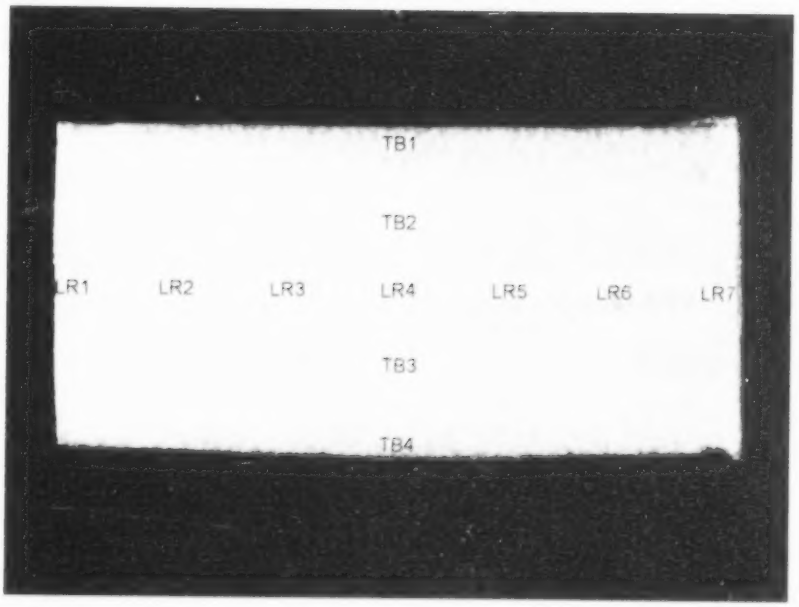


Figure 32. Locations on the exposed injection face of the Y_2NO_3-soaked block for Xe analyses by ESEM-EDS.

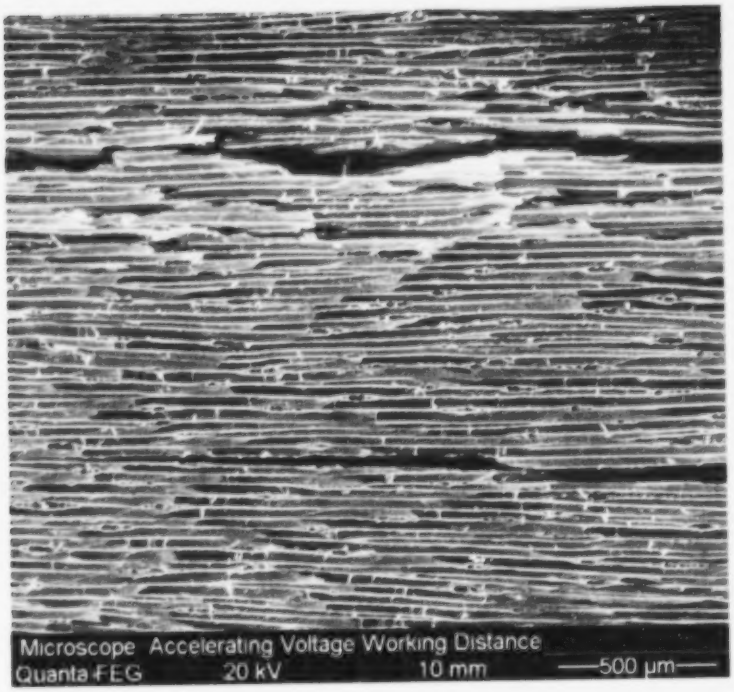


Figure 33. ESEM micrograph of a late-grey block after soaking in 10% $AgNO_3$ for five minutes.

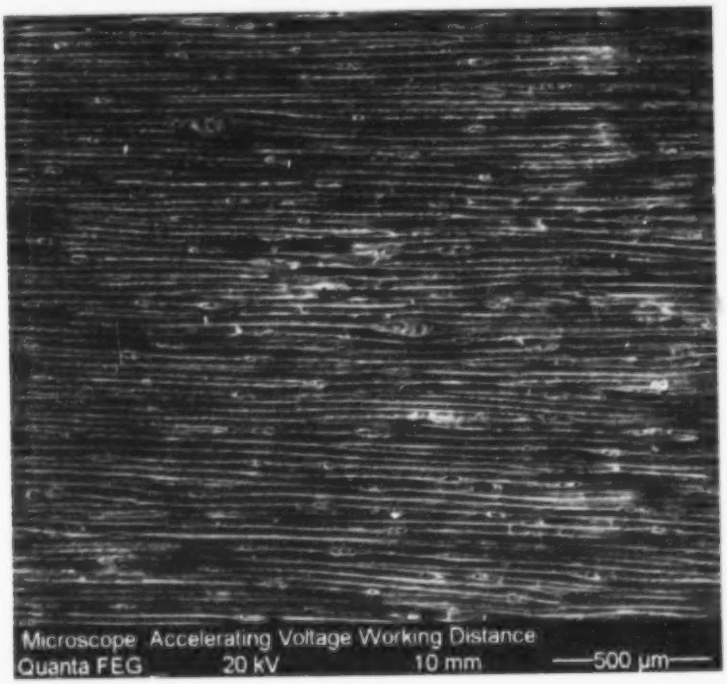


Figure 34. ESEM micrograph of a green block after soaking in 10% $AgNO_3$ for five minutes.

Figure 35. A close-up ESEM image of a late-grey block after soaking in 10% $AgNO_3$ for five minutes.

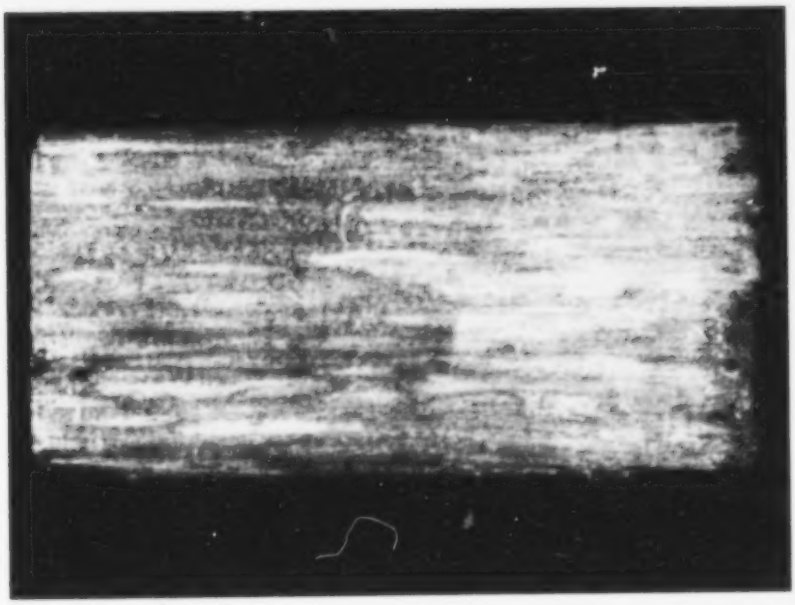


Figure 36. A late-grey block after presteaming and soaking in 10% $AgNO_3$ for 10 minutes.

7 Appendices

Appendix 1. Properties of the green, early-grey and late-grey LPP TMP pulps.

	Green			Early-grey			Late-grey		
Unscreened CSF (mL)	79	100	178	75	95	150	67	107	167
Specific Energy (MJ/kg)	11.5	9.9	8.0	11.3	9.8	8.2	12.8	10.8	8.8
Screened CSF (mL)	86	112	175	79	106	151	75	108	172
Reject (% o.d. pulp)	0.0	0.1	0.1	0.1	0.1	0.1	0.0	0.0	0.1
Apparent Sheet Density (kg/m^3)	374	354	319	361	345	321	361	340	310
Burst Index ($kPa \cdot m^2/g$)	2.6	2.3	2.0	2.7	2.2	2.0	2.5	2.3	1.9
Tensile Index ($N \cdot m/g$)	43	37	34	42	39	34	42	36	33
Stretch (%)	1.65	1.47	1.45	1.73	1.60	1.49	1.85	1.56	1.59
Tear Index ($mN \cdot m^2/g$)	7.7	7.9	8.0	7.5	7.9	8.3	7.8	8.7	9.0
ISO Brightness (%)	56.6	57.4	57.4	50.2	51.2	51.5	50.6	51.4	50.5
ISO Opacity (%)	95.7	94.5	94.2	97.3	96.8	96.5	98.0	97.5	97.0
Scattering Coefficient (cm^2/g)	579	548	535	537	527	524	641	618	580
Sheffield Roughness (SU)	184	211	248	217	244	259	263	273	277
R14 (%)									
R14/28 (%)	8.7	9.6	11.8	10.9	11.1	12.4	10.9	12.6	15.0
R28/48 (%)	29.9	30.9	32.1	30.3	31.1	31.5	29.1	29.8	30.3
R 48/100 (%)	18.5	18.2	18.5	18.3	18.8	18.9	16.9	16.6	16.3
R 100/200 (%)	10.0	10.0	9.8	9.7	10.0	9.9	9.4	9.2	8.8
Pass 200 (%)	5.8	5.1	4.7	5.4	5.3	4.9	5.3	5.4	4.4
R-48 Fraction (%)	27.2	26.1	23.2	25.4	23.9	22.5	28.4	26.6	25.2
W. Weighted Average	57.1	58.7	62.4	59.6	60.9	62.8	56.9	58.9	61.6

Fibre Length (mm)	2.16	2.21	2.25	2.26	2.28	2.33	2.35	2.42	2.50
L. Weighted Average Fibre Length (mm)	1.52	1.56	1.60	1.56	1.57	1.63	1.60	1.67	1.72
Arithmetic Average Fibre Length (mm)	0.61	0.63	0.65	0.60	0.60	0.63	0.59	0.62	0.63

Appendix 2. Properties of blends of the green and late-grey LPP TMP pulps.

	75/25 (wt./wt. o.d.) Green/Late-grey			50/50 (wt./wt. o.d.) Green/Late-grey			25/75 (wt./wt. o.d.) Green/Late-grey		
Unscreened CSF (mL)	78	136	192	58	81	157	72	101	150
Specific Energy (MJ/kg)	11.2	9.3	7.7	12.0	10.5	82	12.4	10.8	9.0
Screened CSF (mL)	82	137	195	68	90	163	74	103	144
Reject (% o.d. pulp)	0.0	0.1	0.2	0.0	0.0	0.1	0.0	0.1	0.1
Apparent Sheet Density (kg/m^3)	371	335	304	399	364	309	354	329	312
Burst Index ($kPa \cdot m^2/g$)	2.7	2.4	2.0	2.7	2.5	2.0	2.6	2.2	1.8
Tensile Index ($N \cdot m/g$)	44	40	35	45	41	35	42	37	31
Stretch (%)	1.69	1.84	1.71	1.85	1.62	1.62	1.62	1.53	1.36
Tear Index ($mN \cdot m^2/g$)	8.1	9.1	9.3	7.3	8.1	8.7	7.9	8.3	8.5
ISO Brightness (%)	54.0	54.9	54.2	54.8	55.2	54.5	50.4	53.5	52.8
ISO Opacity (%)	96.4	94.9	94.5	96.2	95.8	94.9	97.4	96.2	96.1
Scattering Coefficient (cm^2/g)	571	528	518	591	571	532	577	568	563
Sheffield Roughness (SU)	177	214	276	161	209	249	195	228	268
R14 (%)									
R14/28 (%)	13.6	16.2	16.5	9.8	10.6	13.3	10.2	11.7	12.1
R28/48 (%)	30.6	32.1	32.6	30.1	30.5	31.7	30.5	30.6	31.7
R 48/100 (%)	16.4	16.7	15.8	18.4	17.8	17.6	18.3	18.3	18.2
R 100/200 (%)	9.0	8.8	8.5	9.3	9.8	9.3	10.0	10.2	10.0
Pass 200 (%)	5.4	5.0	4.3	6.3	5.7	5.1	6.5	5.7	5.3
R-48 Fraction (%)	25.0	21.3	22.4	26.2	25.7	23.0	24.6	23.4	22.7
W. Weighted Average	60.6	65.0	64.9	58.3	58.8	62.5	59.0	60.6	62.0

Fibre Length (mm)	2.27	2.29	2.35	2.29	2.28	2.35	2.29	2.36	2.41
L. Weighted Average Fibre Length (mm)	1.54	1.55	1.58	1.54	1.59	1.64	1.63	1.69	1.73
Arithmetic Average Fibre Length (mm)	0.58	0.58	0.59	0.59	0.61	0.63	0.63	0.66	0.66

Appendix 3. Properties of the 50/50 green/early-grey, air-dried green, and air-dried early-grey TMP pulps.

	50/50 (wt./wt. o.d.) Green/Early-grey			Air-dried, Green			Air-dried, Early-grey		
Unscreened CSF (mL)	64	115	179	81	132	196	59	86	150
Specific Energy (MJ/kg)	11.9	9.7	8.0	11.1	9.3	7.7	11.3	9.8	7.9
Screened CSF (mL)	66	119	188	79	135	194	66	96	160
Reject (% o.d. pulp)	0.0	0.0	0.1	0.0	0.0	0.1	0.0	0.1	0.1
Apparent Sheet Density (kg/m^3)	375	333	302	354	331	285	368	351	313
Burst Index ($kPa \cdot m^2/g$)	2.8	2.5	2.1	2.5	2.1	1.7	2.7	2.3	2.0
Tensile Index (N·m/g)	47	40	35	41	36	29	44	41	36
Stretch (%)	1.79	1.71	1.60	1.64	1.60	1.39	1.64	1.68	1.62
Tear Index ($mN \cdot m^2/g$)	8.0	8.7	9.7	8.1	8.2	8.3	7.8	8.2	8.5
ISO Brightness (%)	53.3	54.3	53.6	56.5	57.6	56.8	51.6	52.3	52.8
ISO Opacity (%)	96.3	95.0	94.4	95.4	94.4	93.0	97.1	96.5	96.1
Scattering Coefficient (cm^2/g)	550	509	493	555	544	504	567	540	538
Sheffield Roughness (SU)	192	222	279	190	228	295	214	232	278
R14 (%)									
R14/28 (%)	11.8	14.2	16.6	9.4	10.4	12.2	10.1	11.5	12.7
R28/48 (%)	30.5	32.0	32.8	31.6	32.2	33.8	30.3	31.6	32.0
R 48/100 (%)	17.2	16.7	16.3	18.6	18.0	18.2	17.8	18.0	17.9
R 100/200 (%)	9.1	8.8	8.5	10.1	9.7	9.7	9.8	9.6	9.5
Pass 200 (%)	6.4	5.2	4.7	6.3	5.4	5.0	6.4	5.8	5.1

R-48 Fraction (%)	25.0	23.3	21.2	24.0	24.3	21.1	25.7	23.5	22.7
W. Weighted Average Fibre Length (mm)	59.5	62.8	65.7	59.6	60.6	64.2	58.2	61.1	62.7
L. Weighted Average Fibre Length (mm)	2.25	2.36	2.39	2.16	2.20	2.25	2.26	2.28	2.37
Arithmetic Average Fibre Length (mm)	1.57	1.67	1.71	1.51	1.57	1.61	1.56	1.57	1.65
	0.60	0.64	0.66	0.60	0.64	0.66	0.61	0.61	0.63

Appendix 4. Properties of TMP produced at conventional and low intensity.

Early grey-stage (conventional refining intensity)

Refining stage	1st stage	2nd stage	3rd stage	3rd stage	3rd stage	3rd stage
Feed rate (kg/min)	2.13	2.52	2.52	2.52	2.52	2.52
Refiner speed (rpm)	1800	1200	1200	1200	1200	1200
Total specific energy (kWh/odt)	396	1570	2202	2799	3376	3978
CSF (mL)	715	459	235	125	61	40
Somerville rejects (%)	37.520	0.655	0.085	0.050	0.030	0.045
Bauer McNett R-14 (%)	40.7	35.0	30.2	27.1	24.0	19.5
Bauer McNett 14/28 (%)	26.9	24.8	25.2	23.1	21.7	21.1
Bauer McNett 28/48 (%)	13.5	14.3	14.4	14.2	14.7	14.9
Bauer McNett 48/100 (%)	5.5	6.7	7.3	7.5	8.6	9.0
Bauer McNett 100/200 (%)	2.7	3.6	4.3	4.8	5.7	6.0
Bauer McNett P-200 (%)	10.8	15.7	18.6	23.3	25.3	29.6
L. weighted avg. fibre length (mm)	2.32	2.05	1.96	1.85	1.79	1.74
Apparent density (g/cm^3)			0.26	0.30	0.35	0.37
Burst index (kPa·m^2/g)			1.68	2.46	3.00	3.51
Tensile index (N·m/g)			30.22	40.83	50.57	56.97
TEA index (mJ/g)			377.14	626.74	793.12	978.52
Tear index (mN·m^2/g)			9.06	9.35	8.37	7.84
Gurley air resistance (sec/100 mL)			NA	23.1	95.5	223.2
Scattering coefficient (m^2/kg)			48.50	55.02	64.50	66.29
Absorption coefficient (m^2/kg)			3.71	4.07	4.95	5.15
ISO brightness (%)			51.78	52.78	53.29	53.50

Early grey-stage (low refining intensity)

Refining stage	1st stage	2nd stage	3rd stage	3rd stage	3rd stage	3rd stage
Feed rate (kg/min)	2.13	2.52	2.52	2.52	2.52	2.52
Refiner speed (rpm)	1800	900	900	900	900	900
Total specific energy (kWh/odt)	396	1595	2212	2818	3371	4017
CSF (mL)	715	476	321	173	103	61
Somerville rejects (%)	37.520	0.990	0.680	0.325	0.270	0.230
Bauer McNett R-14 (%)	40.7	36.8	32.1	31.0	27.3	23.1
Bauer McNett 14/28 (%)	26.9	26.3	25.0	23.3	22.6	21.4
Bauer McNett 28/48 (%)	13.5	14.1	13.1	13.0	13.3	13.2
Bauer McNett 48/100 (%)	5.5	6.1	5.9	6.2	6.9	7.3
Bauer McNett 100/200 (%)	2.7	3.4	3.4	3.8	4.3	4.7
Bauer McNett P-200 (%)	10.8	13.4	20.5	22.8	25.6	30.3
L. weighted avg. fibre length (mm)	2.32	2.07	2.10	1.95	1.97	2.01
Apparent density (g/cm^3)			0.24	0.28	0.29	0.33
Burst index (kPa·m^2/g)			1.40	2.15	2.59	3.07
Tensile index (N·m/g)			27.90	36.43	42.19	47.97
TEA index (mJ/g)			351.13	514.97	671.45	768.63
Tear index (mN·m^2/g)			9.77	9.88	10.01	8.89
Gurley air resistance (sec/100 mL)			NA	10.4	31.8	93.2
Scattering coefficient (m^2/kg)			44.34	49.16	57.11	63.47
Absorption coefficient (m^2/kg)			3.55	3.76	4.28	4.75
ISO brightness (%)			51.05	52.23	53.04	53.68

Green

Refining stage	1st stage	2nd stage	3rd stage	3rd stage	3rd stage	3rd stage
Feed rate (kg/min)	2.56	2.52	2.52	2.52	2.52	2.52
Refiner speed (rpm)	1800	1200	1200	1200	1200	1200
Total specific energy (kWh/odt)	345	1529	2166	2792	3315	3898
CSF (mL)	724	517	291	144	81	41
Somerville rejects (%)	30.340	0.560	0.045	0.020	0.017	0.027
Bauer McNett R-14 (%)	39.4	31.6	28.9	25.1	21.7	14.8
Bauer McNett 14/28 (%)	29.7	27.4	26.7	25.0	24.0	22.9
Bauer McNett 28/48 (%)	13.8	14.4	14.2	13.6	14.1	15.1
Bauer McNett 48/100 (%)	5.6	6.5	6.8	7.3	8.0	9.1
Bauer McNett 100/200 (%)	2.9	3.7	4.0	4.5	5.0	5.8
Bauer McNett P-200 (%)	8.7	16.5	19.5	24.5	27.2	32.4
L. weighted avg. fibre length (mm)	2.26	2.16	1.94	1.98	1.81	1.60
Apparent density (g/cm^3)			0.24	0.30	0.33	0.37
Burst index (kPa·m^2/g)			1.39	2.29	2.67	3.02
Tensile index (N·m/g)			25.76	37.70	45.80	52.83
TEA index (mJ/g)			269.42	535.68	647.18	853.38
Tear index (mN·m^2/g)			8.42	9.63	9.19	8.08
Gurley air resistance (sec/100 mL)			NA	21.1	59.4	197.4
Scattering coefficient (m^2/kg)			45.85	54.53	60.13	65.98
Absorption coefficient (m^2/kg)			1.85	1.96	2.18	2.29
ISO brightness (%)			59.41	61.31	61.65	62.53

Late grey-stage (conventional refining intensity)

Refining stage	1st stage	2nd stage	3rd stage	3rd stage	3rd stage	3rd stage
Feed rate (kg/min)	2.02	2.52	2.52	2.52	2.52	2.52
Refiner speed (rpm)	1800	1200	1200	1200	1200	1200
Total specific energy (kWh/odt)	400	1618	2260	2852	3469	4011
CSF (mL)	700	359	191	100	54	30
Somerville rejects (%)	29.830	0.470	0.050	0.030	0.044	0.090
Bauer McNett R-14 (%)	48.4	29.9	25.4	24.0	19.6	15.1
Bauer McNett 14/28 (%)	25.7	22.9	22.0	20.7	19.6	18.9
Bauer McNett 28/48 (%)	14.3	14.9	14.4	14.5	14.5	15.3
Bauer McNett 48/100 (%)	6.6	7.8	7.8	8.5	9.1	10.1
Bauer McNett 100/200 (%)	3.5	4.9	5.1	6.0	6.7	7.7
Bauer McNett P-200 (%)	1.4	19.6	25.3	26.3	30.6	32.9
L. weighted avg. fibre length (mm)	2.38	2.01	1.84	1.86	1.73	1.57
Apparent density (g/cm³)			0.27	0.31	0.35	0.39
Burst index (kPa·m²/g)			1.75	2.54	2.93	3.34
Tensile index (N·m/g)			29.84	38.78	49.08	53.39
TEA index (mJ/g)			384.94	512.66	782.53	898.38
Tear index (mN·m²/g)			9.31	8.76	7.85	7.33
Gurley air resistance (sec/100 mL)			8.9	39.7	116.7	292.5
Scattering coefficient (m²/kg)			56.14	65.56	72.50	77.08
Absorption coefficient (m²/kg)			3.86	4.33	4.87	5.38
ISO brightness (%)			52.37	53.53	53.82	53.98

Late grey-stage (low refining intensity)

Refining stage	1st stage	2nd stage	3rd stage	3rd stage	3rd stage	3rd stage
Feed rate (kg/min)	2.02	2.52	2.52	2.52	2.52	2.52
Refiner speed (rpm)	1800	900	900	900	900	900
Total specific energy (kWh/odt)	400	1623	2235	2822	3459	4031
CSF (mL)	700	489	301	153	73	59
Somerville rejects (%)	29.830	1.590	0.690	0.325	0.215	0.195
Bauer McNett R-14 (%)	48.4	37.5	33.5	30.2	25.1	22.0
Bauer McNett 14/28 (%)	25.7	23.4	22.7	21.0	20.3	19.2
Bauer McNett 28/48 (%)	14.3	13.2	13.1	13.4	13.9	13.6
Bauer McNett 48/100 (%)	6.6	6.1	6.5	7.1	8.0	8.1
Bauer McNett 100/200 (%)	3.5	3.6	4.2	4.9	5.8	5.9
Bauer McNett P-200 (%)	1.4	16.3	20.0	23.5	26.9	31.3
L. weighted avg. fibre length (mm)	2.38	2.20	2.09	2.00	1.88	1.85
Apparent density (g/cm³)			0.23	0.28	0.33	0.36
Burst index (kPa·m²/g)			1.34	2.09	2.84	2.70
Tensile index (N·m/g)			23.28	35.35	45.65	47.11
TEA index (mJ/g)			259.17	492.98	727.49	821.82
Tear index (mN·m²/g)			9.20	9.66	8.49	8.23
Gurley air resistance (sec/100 mL)			NA	15.5	89.6	147.6
Scattering coefficient (m²/kg)			49.25	58.53	66.13	69.52
Absorption coefficient (m²/kg)			3.82	4.21	4.81	5.02
ISO brightness (%)			50.37	52.11	52.81	53.02

Appendix 5. Properties of TMP from pretreated chips.

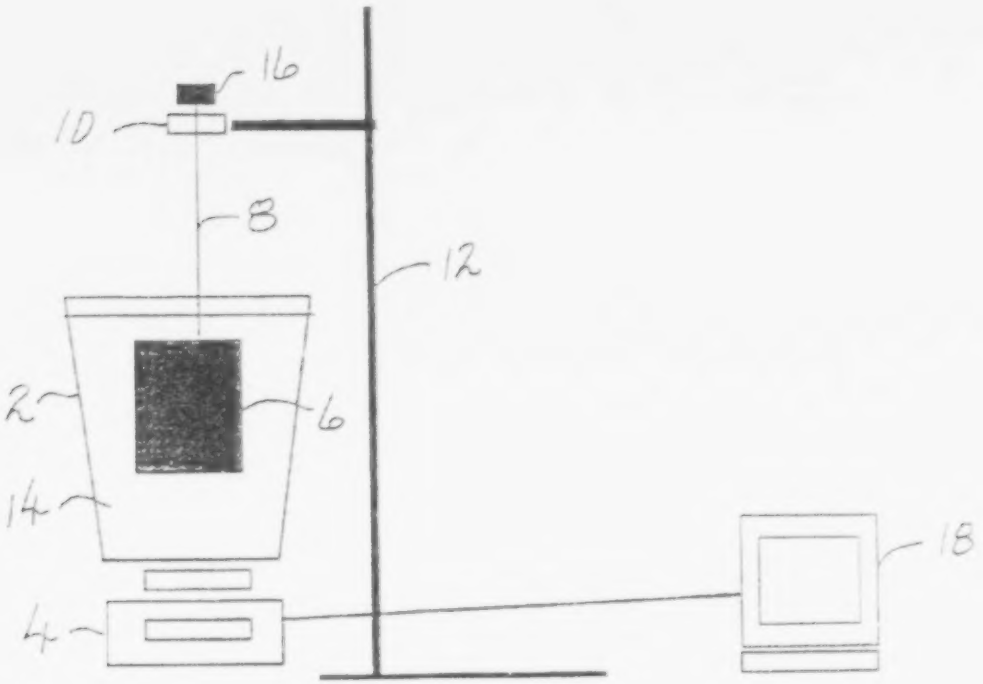
Late grey-stage with water impregnation (low intensity)

Refining stage	1st stage	2nd stage	3rd stage	3rd stage	3rd stage	3rd stage
Feed rate (kg/min)	1.43	2.52	2.52	2.52	2.52	2.52
Refiner speed (rpm)	1800	900	900	900	900	900
Total specific energy (kWh/odt)	502	1716	2328	2925	3512	4133
CSF (mL)	701	446	360	180	113	60
Somerville rejects (%)	22.420	1.330	0.690	0.250	0.170	0.150
Bauer McNett R-14 (%)	40.6	31.8	38.6	32.2	27.6	22.8
Bauer McNett 14/28 (%)	25.3	25.1	23.4	21.6	19.4	18.6
Bauer McNett 28/48 (%)	13.8	14.1	14.6	14.4	13.8	13.8
Bauer McNett 48/100 (%)	6.1	6.7	7.6	7.9	7.9	8.5
Bauer McNett 100/200 (%)	3.1	3.8	4.3	4.8	4.8	5.4
Bauer McNett P-200 (%)	11.0	18.6	11.5	19.2	26.6	31.0
L. weighted avg. fibre length (mm)	2.21	2.03	2.18	2.08	2.06	1.88
Apparent density (g/cm^3)			0.22	0.27	0.31	0.34
Burst index (kPa·m^2/g)			1.22	1.84	2.61	2.91
Tensile index (N·m/g)			22.26	32.46	41.39	50.60
TEA index (mJ/g)			209.97	447.61	607.07	847.19
Tear index (mN·m^2/g)			8.92	9.91	9.59	8.17
Gurley air resistance (sec/100 mL)			NA	8.0	41.7	138.1
Scattering coefficient (m^2/kg)			46.38	52.72	61.14	69.40
Absorption coefficient (m^2/kg)			3.74	3.96	4.25	4.93
ISO brightness (%)			49.65	51.07	52.85	53.19

Late grey-stage with sulfite impregnation (low intensity)

Refining stage	1st stage	2nd stage	3rd stage	3rd stage	3rd stage	3rd stage
Feed rate (kg/min)	1.62	2.52	2.52	2.52	2.52	2.52
Refiner speed (rpm)	1800	900	900	900	900	900
Total specific energy (kWh/odt)	367	1586	2188	2790	3372	4008
CSF (mL)	709	540	417	252	128	77
Somerville rejects (%)	34.340	1.270	0.440	0.110	0.090	0.070
Bauer McNett R-14 (%)	43.1	40.5	35.0	32.8	29.6	26.3
Bauer McNett 14/28 (%)	24.8	25.9	23.7	22.1	21.4	20.5
Bauer McNett 28/48 (%)	12.9	14.3	13.4	13.2	13.3	13.5
Bauer McNett 48/100 (%)	5.1	6.5	6.5	6.8	7.5	8.1
Bauer McNett 100/200 (%)	2.5	3.5	3.8	4.2	4.7	5.3
Bauer McNett P-200 (%)	11.6	9.2	17.6	21.0	23.5	26.4
L. weighted avg. fibre length (mm)	2.38	2.20	2.17	2.24	2.16	1.98
Apparent density (g/cm³)			0.21	0.26	0.32	0.35
Burst index (kPa·m²/g)			1.21	2.13	2.82	3.27
Tensile index (N·m/g)			24.48	35.40	45.38	53.75
TEA index (mJ/g)			254.66	462.97	730.31	927.52
Tear index (mN·m²/g)			9.08	11.08	9.72	8.41
Gurley air resistance (sec/100 mL)			NA	6.7	31.3	106.5
Scattering coefficient (m²/kg)			43.58	48.47	52.59	58.67
Absorption coefficient (m²/kg)			2.29	2.31	2.49	2.75
ISO brightness (%)			54.49	56.30	56.79	57.38

Appendix 6. Weyerhaeuser chip impregnation test apparatus (2 - H_2O-container, 4 - analytical balance, 6 - covered wire mesh basket containing chips, 10 - clamp, 14 - DI H_2O or an aqueous solution, 16 - stopper, 18 - computer).



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